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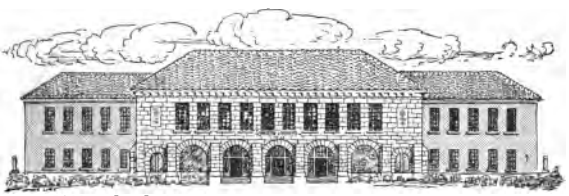

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CHEMICAL EXPERIMENTS

GENERAL AND ANALYTICAL

FOR USE WITH ANY TEXT-BOOK OF CHEMISTRY, OR WITHOUT A TEXT-BOOK

BY

R. P. WILLIAMS

Instructor in Chemistry, English High School, Boston, and Author of "Introduction to Chemical Science," "Laboratory Manual," etc.

BOSTON, U.S.A., AND LONDON
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PREFACE.

THE rapid growth of the laboratory method of teaching chemistry since the author's *Manual* was issued in 1888 has called for a larger and more comprehensive work of a similar sort for high schools, academies, and colleges. To such a call the present book is a response.

In preparing the experiments the author has endeavored, *first*, to select such as are most instructive and best illustrate the subject without being too elaborate; *second*, to arrange them in an order calculated to lead up by the most natural and easiest steps to a knowledge of the science; *third*, to make the subject fascinating by giving just enough information and suggestion to interest the experimenter, and to make him work for the knowledge to be gained. Finally, the author has aimed to make the book simple enough for the dull and slow pupil, and — by the introduction of supplementary and original work — elaborate enough for the most acute. Great care has been taken to give minute and accurate directions for performing the experiments. "Terms" at the end of experiments are for pupils to study outside the laboratory, and are often repeated. They may also be discussed in the laboratory.

Metal analysis has received a somewhat unique treatment. Each metal of a group is first taken separately, and the *analytical* reactions are given. This method shows the pupil at once why a given re-agent is added, and what its effect is. The product is given when the substance is in solution as well as when it is precipitated. The group is next treated in the same way. This method clarifies the subject greatly in the pupil's mind, and is believed to be a valuable departure in teaching analysis.

71037

The new orthography recommended by the Chemical Section of the A. A. A. S. has been adopted, as a step towards uniformity and progress.

It is recommended that wherever it is possible each laboratory period cover the space of two hours (even if there can be only one period per week), and that the last 20 or 30 minutes be taken to question the class rapidly on the work of the day, discuss and explain the use of terms, etc. The books should be inspected after each exercise, or perhaps after each experiment has been written out.

It is by no means necessary to perform every experiment, or to follow the order here given, if the teacher prefers to do otherwise. A diversity of opinion exists among teachers, and a diversity of time and talent among pupils and classes. In no place better than in the laboratory, under a competent instructor, can a "cast-iron rule" be done away with, and variety of arrangement and method be adopted.

The author would be glad to receive criticisms, as well as suggestions as to other methods, or experiments, or different ways of performing experiments. Especially would he be glad to know what it has been difficult, in the matter of directions or principles, for pupils to understand.

The author desires to acknowledge valuable suggestions in the preparation of this work from the following persons: Messrs. J. Y. Bergen, Jr., C. W. Gerould, E. F. Holden, A. S. Perkins, N. S. French, H. I. Lord, E. S. Chapin; and Misses D. M. Stickney, M. L. Foster, and others.

CAMBRIDGE,
December 1, 1895.

INDIVIDUAL APPARATUS.

Each pupil should be provided with the apparatus given below, but in cases where great economy must be exercised different pupils may, by working at different times, use the same set. The author has selected apparatus specially adapted, as to exact dimensions, quality, and cheapness, for performing in the best way the experiments herein described, and sets or separate pieces of this, together with other apparatus and chemicals, can be had of the L. E. Knott Apparatus Co., 14 Ashburton Place, Boston, to which firm teachers are referred for catalogs.

- 4 re-agent bottles, 250 cc. glass stoppers, blown labels: NH_4OH, HCl, HNO_3, H_2SO_4.
- 1 pneumatic trough.
- 1 Hessian crucible.
- 2 beakers.
- 2 pieces wire gauze.
- 1 piece platinum wire.
- 1 mouth blowpipe.
- 6 pieces glass tubing.
- 4 pieces hard glass tubing.
- 1 test-tube brush.
- 1 small tube brush.
- 1 doz. test tubes.
- 4 cork stoppers, for tubes.
- 1 fish-tail attachment for Bunsen burner.
- 1 blowpipe attachment with rest for Bunsen burner.
- 1 piece sheet copper.
- 1 glass retort.
- 1 tumbler.
- 1 piece cobalt glass.
- 1 horn spatula.
- 1 sheet litmus paper.
- 200 filter papers.
- 1 bunch splints.
- 1 sheet turmeric paper.
- 1 camel's-hair brush.
- 1 magnet.
- 4 wide-mouthed bottles.
- 1 2-holed rubber stopper to fit above.
- 4 pieces window-glass.
- 1 glass funnel.
- 1 porcelain evap. dish.
- 1 asbestos board.
- 1 sand bath.
- 1 pair iron forceps.
- 1 triangular file.
- 1 round file.
- 1 piece copper wire.
- 1 piece lead wire.
- 1 piece zinc wire.
- 1 wooden test-tube holder.
- 1 wire test-tube rack.
- 1 thistle tube.
- 1 Bohemian flask.
- 1 2-holed rubber stopper to fit flask.
- 1 Bunsen burner.
- 1 iron ring stand.
- 2 ft. rubber tubing.
- 1 ft. " "
- 1 metric ruler.
- 1 graduate.
- 1 small leaden dish.

GENERAL APPARATUS.

The requirements of general apparatus for a laboratory are too numerous and varied to be given here. A few of the pieces in more general demand for experiments in this book are as follows:

Glass-stoppered bottles.
Graduates.
Funnels.
Fruit jars (for making solutions).
Scales, with metric weights.
Ignition tubes.
Beakers.
Filter-stands.
Glass and rubber tubing.
Steel glass-cutters.
Steel wire-cutters.
Mortars and pestles.
Flasks.
Filter papers, etc., etc.

CHEMICALS.

ESTIMATE FOR A CLASS OF TWENTY IN GENERAL CHEMISTRY AND ANALYTICAL REACTIONS, NOT INCLUDING GENERAL REACTIONS AND SUPPLEMENTARY WORK.

Acid, acetic, $HC_2H_3O_2$	4 lb.	Arsenopyrite, FeAsS	½ lb.
" boric, H_3BO_3	½ "	Barium chlorid, $BaCl_2$	½ "
" hydrochloric, HCl	25 "	" hydroxid, $Ba(OH)_2$	½ "
" hydrofluoric, HF	½ "	Bismuth oxychlorid, BiOCl	½ "
" hydrofluosilicic, H_2SiF_6	½ "	Calcium chlorid, $CaCl_2$	½ "
" nitric, HNO_3	12 "	" fluorid, powdered, CaF_2	½ "
" oxalic, $H_2C_2O_4$	½ "	" hypochlorite, $CaCl_2O$	½ "
" phosphoric, H_3PO_4	½ "	" sulfate, $CaSO_4$	½ "
" sulfuric, H_2SO_4	18 "	" oxid, CaO	2 "
" tartaric, $H_2C_4H_4O_6$	½ "	Carbon bisulfid, CS_2	½ "
Alcohol, C_2H_5OH	1 qt.	Charcoal, animal, gran., C	1 "
Alum, $K_2Al_2(SO_4)_4$	1 lb.	" " powdered, C	1 "
Aluminum chlorid, $AlCl_3$	½ "	" wood, " C	1 "
Ammonium carbon., $(NH_4)_2CO_3$	1 "	" " C	20 pieces
" chlorid, NH_4Cl	2 "	Cobalt chlorid, $CoCl_2$	½ lb.
" hydroxid, NH_4OH	20 "	" nitrate, $Co(NO_3)_2$	½ "
" nitrate, NH_4NO_3	1 "	Cochineal	½ "
" oxalate, $(NH_4)_2C_2O_4$	½ "	Copper, turnings, Cu	8 "
" sulfate, $(NH_4)_2SO_4$	½ "	" sheet, Cu	½ "
Antimony cryst., Sb	½ "	" chlorid, $CuCl_2$	1 "
" chlorid, $SbCl_3$	½ "	" nitrate, $Cu(NO_3)_2$	½ "
Arsenious oxid, As_4O_6	1 oz.	" oxid, CuO	1 "

Copper sulfate, $CuSO_4$ ..... ½ lb.
Ether, $(C_2H_5)_2O$ ..... ½ "
Gold leaf, Au ..... 1 bk.
Indigo ..... ½ lb.
Iodin, I ..... 2 oz.
Iron, fine turnings, Fe ..... 1 lb.
" by hydrogen, Fe ..... ½ "
" perchlorid, $FeCl_3$ ..... 1 "
" protosulfid, FeS ..... 5 "
" pyrite, FeS_2 ..... ½ "
" sulfate, $FeSO_4$ ..... 1 "
Lead, sheet, Pb ..... 1 "
" wire, ⅛ in. diam., Pb ..... 12 ft.
" acetate, $Pb(C_2H_3O_2)_2$ ..... ½ lb.
" chlorid, $PbCl_2$ ..... ½ "
" nitrate, $Pb(NO_3)_2$ ..... 2 "
" protoxid, PbO ..... ½ "
" tetroxid (red), Pb_3O_4 ..... ½ "
Litmus, cubes ..... 1 oz.
Marble chips, $CaCO_3$ ..... 3 lb.
Magnesium ribbon, Mg ..... 10 ft.
" chlorid, $MgCl_2$ ..... ½ lb.
" sulfate, $MgSO_4$ ..... ½ "
Manganese chlorid, $MnCl_2$ ..... ½ "
" dioxid, gran., MnO_2 .. ½ "
" " powd., MnO_2 1½ "
Mercury, Hg ..... ½ "
" bichlorid, $HgCl_2$ ..... ¼ "
" protochlorid, HgCl ..... ½ "
" pernitrate, $Hg(NO_3)_2$ .... ½ "
" protonitrate, $HgNO_3$ .... ½ "
" oxid, HgO ..... 1 "
Nessler's sol. ..... ½ "
Phosphorus, P ..... 2 oz.
Picture wire ..... 1 roll
Platinum chlorid, sol., $PtCl_4$ ..... 1 oz.
Potassium, metallic, K ..... ½ "
" bromid, KBr ..... ½ lb.
" carbonate, K_2CO_3 ..... 1½ "
" chlorid, KI ..... ¼ "
" chlorate, $KClO_3$ ..... 2 "
" chromate, K_2CrO_4 .... ½ "
" cyanid, KCN ..... ½ "
Potassium, dichromate, $K_2Cr_2O_7$ 1 lb.
" ferrocy., $K_4Fe(CN)_6$ .. 1 "
" ferricy., $K_3Fe(CN)_6$ .. ¼ "
" iodid, KI ..... 1 "
" hydroxid, KOH ..... 1 "
" nitrate, KNO_3 ..... 1 "
" nitrite, KNO_2 ..... ½ "
" permangan., $KMnO_4$ ½ "
" sulfocyanid, KSCN .... ½ "
" tartrate, $K_2C_4H_4O_6$ .... ½ "
Silica, powdered, SiO_2 ..... ½ "
Silver nitrate, $AgNO_3$ ..... 4 oz.
Sodium, metallic, Na ..... ½ "
" acetate, $NaC_2H_3O_2$ ..... ½ lb.
" arsenite, HNa_2AsO_3 ..... ½ "
" bicarbonate, $HNaCO_3$ .... ½ "
" bisulfate, $HNaSO_4$ ..... ½ "
" borate, $Na_2B_4O_7$ ..... ½ "
" carbonate, Na_2CO_3 ..... 3 "
" chlorid, NaCl ..... 4 "
" hydroxid (caust.), NaOH 3 "
" nitrate, $NaNO_3$ ..... 2 "
" nitrite, $NaNO_2$ ..... ½ "
" oxalate, $Na_2C_2O_4$ ..... ½ "
" phosphate, HNa_2PO_4 ..... 1 "
" sulfate, Na_2SO_4 ..... 1 "
" sulfid, Na_2S ..... ½ "
" sulfite, Na_2SO_3 ..... ½ "
" thiosulfate, $Na_2S_2O_3$ ..... 1 "
Starch, $C_6H_{10}O_5$ ..... ½ "
Strontium chlorid, $SrCl_2$ ..... ½ "
Sugar, $C_{12}H_{22}O_{11}$ ..... 1 "
Sulfur, brimstone, S ..... 2 "
" flowers, S ..... 1 "
Tin bichlorid, $SnCl_2$ ..... ½ "
Turmeric paper ..... 1 sheet
Turpentine, $C_{10}H_{16}$ ..... 1 oz.
Zinc, gran., Zn ..... 3 lb.
" wire, ⅛ in. diam. ..... 12 ft.
" (arsenic-free) ..... 1 lb.
" chlorid, $ZnCl_2$ ..... ½ "
" nitrate, $Zn(NO_3)_2$ ..... ½ "
" Litmus paper ..... ½ quire

SOLUTIONS, ETC.

In making solutions for metal analysis, chlorids of the metals are usually taken (except in Group I, in whlch nitrates are used, the chlorids being insoluble). From 25g to 50g of solids are usually dissolved in 500cc of distilled water, and filtered if necessary. Many are sat., as $Ca(OH)_2$. Substances for analysis, as well as acids and other re-agents, should be C. P. Concentrated acids are used unless the dilute are called for.

A few exceptional sols. are given below:

HNa_2PO_4,	40.	$(NH_4)_2CO_3$,	100.	$(NH_4)_2SO_4$,	5.
KCy,	50.	KNO_2,	50.	NH_4Cl,	60.
$K_2Cr_2O_7$,	25.*	KOH,	60.	NaOH,	60.

Dilute acids are made by mixing one volume of concentrated acid with four volumes of water.

NH_4OH. If NH_4OH of 26° is obtained, it should be diluted with three times its volume of water before using.

$HgNO_3$ is best prepared by putting into a large e.d. say 25g of Hg, and adding to it a mixture of 25cc HNO_3 and 25cc of H_2O, and letting it stand till action stops, then adding 400cc or 500cc of H_2O. More Hg should be used than will dissolve, and some should be left in the bottle.

$PbCl_2$ is only slightly soluble in water. A saturated sol. should be used.

$BiCl_3$ is prepared by adding water and HCl to oxychlorid of bismuth, BiOCl. First add the H_2O, then acidulate it with the acid, say 1 vol. of HCl to 20 of H_2O, it being insol. in H_2O alone.

$AsCl_3$. Add H_2O to sodium arsenite HNa_2AsO_3, then add HCl with stirring till effervescence stops.

$SbCl_3$. Pour a little butter of antimony, $SbCl_3$, into H_2O, then acidulate with HCl.

$SnCl_2$. This must also be acidulated with HCl, and a little Sn should be left in the bottom of the bottle.

$CrCl_3$. This may be made from $K_2Cr_2O_7$ sol. by adding some HCl (say $\frac{1}{10}$) and a little alcohol, boiling some time and letting it stand, when it turns from red to green.

$(NH_4)_2S$. Pass H_2S gas into dilute NH_4OH till the sol. gives no ppt. with $MgSO_4$ sol., then filter. It should be nearly colorless.

$(NH_4)_2S_x$ (ammonium polysulfid). Stir sulfur flowers in the $(NH_4)_2S$, as above prepared, then filter. It is yellow.

Cochineal sol. Grind up a handful of cochineal in a mortar, and add water, stir, and filter.

Litmus sol. Pulverize litmus cubes, and add water.

Indigo sol. (sulfindigotic acid). Slowly mix and stir 5g indigo with 25cc H_2SO_4 (or better $H_2S_2O_7$, fuming sulfuric acid) in a beaker immersed in cold water. Cover the beaker, and after 48 hours add 500cc H_2O, stir and filter.

Many substances in sol. easily break up on standing, and hence should be freshly prepared.

For acid radicals compounds of Na or K are commonly used, as they are soluble.

RULES AND SUGGESTIONS FOR PUPILS.

Pupils should read these rules carefully, and should refer to them whenever in doubt on any point.

1. Each pupil should provide himself with a towel, to keep his bench clean, and any apron or other clothing desired for protection in the laboratory. A long linen duster or duck coat reaching to the feet is the best protection. Without something of the sort clothing may be injured at any moment.

2. The bench at which the pupil works must be left *clean and dry* after every laboratory exercise. Wipe off a ring-stand, lamp, or other apparatus on which a re-agent has fallen, wipe out a p.t. after using it, and keep re-agent bottles, apparatus, books, and lockers clean.

3. Pupils are held responsible for apparatus, and should replace anything that is lost or broken.

4. In experimenting have your apparatus neatly arranged without artificial props, wedges, or uncouth-looking material. Have every d.t. and stopper fit tightly to prevent leakage of gas. If a gas generates well, but does not pass into the rec., there is some leakage, due probably to loose bearings.

5. In heating a t.t. on the r.s. hold the lamp in the hand, move it slowly, and now and then take it away from the tube for a moment, to prevent melting and breaking the latter.

6. Mixtures of solids should be made on paper. *Be careful not to mix chemicals or re-agents except as directed.*

7. To shake the contents of a t.t. cover its mouth with the thumb (Exp. 4, Fig. 10), or the hand, or a stopper, *hold it away from the bench*, and shake it vigorously.

8. Never put down a stopper when using a re-agent bottle, but hold it between the first and second fingers (Exp. 6, Fig. 11), and replace it when you put down the bottle. Do not pour back any excess of a re-agent from a t.t. or other rec. into a re-agent bottle, and do not dip a stirring rod into a re-agent bottle.

9. In pouring a liquid into a t.t. (or graduate) hold the latter on a level with the eye, and towards the light, so as to see any change. The thumb-nail may be placed at the upper limit to which it is desired the liquid should reach. When much heat is liberated, as when sulfuric acid is poured into water, a tube-holder should be used; paper folded several times serves for this purpose.

10. Pour only liquids, fine powders, or soluble salts into the sinks, *always opening the faucet first to let the water run.* Other solids should be thrown into the jars. *Great care must be taken not to clog the discharge-pipes.*

11. Have flasks and tubes perfectly dry on the outside before applying heat. Tubes of thick glass, if they contain no liquid, should be heated gradually at

first by moving them alternately into and out of the flame. Before lighting the gas of a Bunsen burner, open the jet to its full capacity. If the gas "burns at the base," extinguish and re-light it.

12. Re-agents for general use must not be taken to the individual's bench, but left at the side-table, and nothing must be put into a pupil's locker except what belongs there.

13. The pupil should, before entering the laboratory, make as thorough preparation for experimenting, by studying a text-book, as he would for a recitation. In experimenting follow the directions as closely as possible. Read an exp. through before performing any part of it. *Ask an explanation of anything you do not understand. Observe very carefully everything that takes place, and try to ascertain what is important in the experiment. Note especially the order in which phenomena occur.*

14. Read several times the "Model for taking Notes." Begin to write on the page opposite the experiment. Both name and symbolize substances once in each exp., as "Manganese dioxid, MnO_2"; afterwards in the same exp. use either name or symbol. In writing equations employ only symbols, without names or weights. Indicate gases in equations by a bracket above the symbol, as $\widetilde{NH_3}$; ppts. by a bracket beneath, as $\underset{\sim}{PbCl_2}$.

15. In memorizing experiments learn the names, symbols, processes, and products, but not the quantities of chemicals used.

16. Make a special point of keeping a neat, accurate, and full record of your observations, and what they teach, but do not write in your notes irrelevant matter which may have been read in the text-book, but which the experiment does not show, except in certain cases for explanation.

17. No notes should be written in this book outside of the laboratory without special permission. Note-books should be left in the laboratory at the end of the hour, and no other books should be used in connection with the experiments.

18. Write your name and the division to which you belong distinctly across the front edge of the book in Roman letters. Books should be covered with heavy paper.

19. For burns put some Na_2CO_3 (dried) or $HNaCO_3$ on a handkerchief, moisten it, and bind it on the part affected. If this is done in season no blister will form, and the pain will soon cease.

TAKING NOTES.

It is of the greatest importance that pupils should make accurate observations, and take correct and original notes.

These notes should embody the following points, and no experiment should be accepted unless it does contain them: 1. CHEMICALS (names and symbols); 2. APPARATUS, ADJUSTMENT, etc.; 3. OBSERVATIONS and RESULTS, such as the state (solid, liquid, or gaseous) of the product, its color, odor (if a gas), increase of temperature, etc.; 4. EXPLANATION of each change, with equation wherever that is possible. *1, 2, should be stated very briefly*, not in separate parts, but as in the model below, *and without copying from the page of directions. The main stress should be put on 3 and 4.* Questions, calls for observations, equations, etc., in the directions are suggestive only, and not intended to indicate the complete notes. Methods vary, but the author prefers a plan similar to that outlined below. If this is followed, pupils must study the model carefully. The author has a brief and accurate method for recording Metal Analysis, which he will be glad to indicate to any teacher desiring it.

MODEL FOR NOTES.

PREPARATION OF OXYGEN.

I mixed about 5ᵍ of white crystalline potassium chlorate, $KClO_3$, with not quite as much black, powdered manganese dioxid, MnO_2, put the mixture into a t.t., adjusted a d.t., hung it to a r.s., and arranged so as to collect the gas in recs. over water in a p.t. I applied heat moderately at first, and afterwards more strongly.

I noticed the water in the d.t. began to move forward, as if pushed by a gas, and soon bubbles rose to the top of the rec. These became larger, and rose more rapidly as the heat increased. They gradually drove the water out of the rec. As they burst they threw off a fine gray cloud, which I decided was a solid, as it soon settled, beginning to disappear at the top first. It may have been very fine MnO_2, etc., carried over mechanically in the bubbles of oxygen. An almost colorless gas remained. On heating the mixture in the tube I also noticed slight explosions which threw up particles of MnO_2. These seemed to be caused by the rapid separation of the gas under the MnO_2. The equation which stands for the reaction is

$$KClO_3 = KCl + \overset{\frown}{3O}.$$

Every molecule of solid potassium chlorate breaks up into a molecule of solid potassium chlorid and 3 atoms of gaseous oxygen. The O, occupying much volume, escapes through the d.t.

At the end of the experiment there is in the t.t....., etc.

The back action in the d.t. is caused by....., etc.

TABLE OF COMMON ELEMENTS.

ARRANGED ACCORDING TO ELECTRO-CHEMICAL ORDER.

	NAME.	SYMBOL.	AT. WT.	VAPOR DENSITY.	STATE.
Negative or Non-Metallic Elements. Acid-Forming with H (usually OH).	Oxygen	O″	16	16	G
	Sulfur	S″, IV (VI)	32	32	S
	Nitrogen	N‴, V, etc.	14	14	G
	Fluorin	F′, (V)	19		G
	Chlorin	Cl′, (V)	35.5	35.5	G
	Bromin	Br′, (V)	80	80	L
	Iodin	I′, (V)	127	127	S
	Phosphorus	P‴, V	31	62	S
	Arsenic	As‴, V	75	150	S
	Chromium	Cr″, ‴, VI	52		S
	Boron	B‴	11		S
	Carbon	C(″), IV	12		S
	Antimony	Sb‴, V	120		S
	Silicon	Si IV	28		S
	Hydrogen	H′	1	1	G
Positive or Metallic Elements. Base-Forming with OH.	Gold	Au(′), ‴	197		S
	Platinum	Pt(″), IV	197		S
	Mercury	Hg′, ″	200	100	L
	Silver	Ag′	108		S
	Copper	Cu(′), ″	63		S
	Bismuth	Bi‴, (V)	207		S
	Tin	Sn″, IV	117		S
	Cadmium	Cd″	112	56	S
	Cobalt	Co″, (‴)	59		S
	Nickel	Ni″, (‴)	58		S
	Lead	Pb″, (IV)	206		S
	Iron	Fe″, ‴	56		S
	Zinc	Zn″	65	32½	S
	Manganese	Mn″, (‴)	55		S
	Aluminum	Al‴	27		S
	Magnesium	Mg″	24		S
	Calcium	Ca″	40		S
	Strontium	Sr″	87		S
	Barium	Ba″	137		S
	Sodium	Na′	23		S
	Potassium	K′	39		S

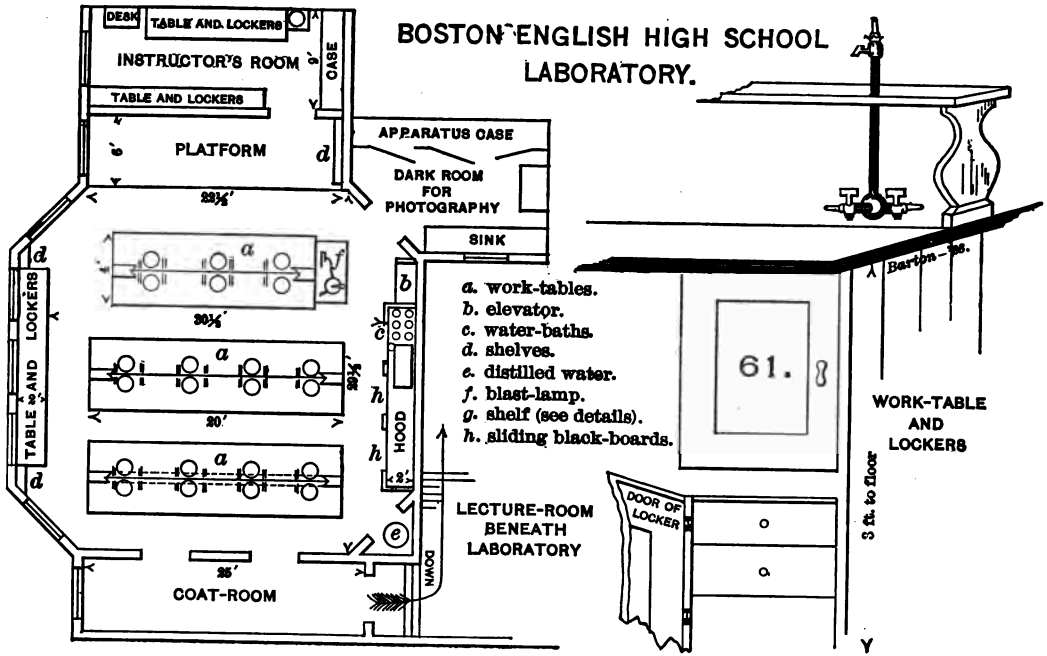
A ( ) indicates a rare valence. Atomic weights are only approximate.

ABBREVIATIONS.

Ac. — acetate, $C_2H_3O_2$.
alk. — alkali or alkaline.
Am. — ammonium, NH_4.
app. — apparatus.
b. — black.
B.B. — before the blowpipe.
B.B.C.C. — before the blowpipe on charcoal.
bl. — blue.
cc. — cubic centimeters.
cm. — centimeters.
col. — color.
conc. — concentrated.
cpd. — compound.
cryst. — crystalline.
Cy. — cyanid, CN.
dil. — dilute.
dis. — dissolve.
dist. — distinguish.
d.s. — deflagrating spoon.
d.t. — delivery tube.
e.d. — evaporating dish.
evap. — evaporate.
ex. — excess.
Exp. — experiment.
fil. — filtrate.
g. — grams.
gel. — gelatinous.
gen. — generator.
gr. — green.
insol. — insoluble.
i.t. — ignition tube.
M. — metal (valence 1).
o.f. — oxidizing flame.
Ox. — oxalate.
ppd. — precipitated.
ppg. — precipitating.
ppn. — precipitation.
ppt. — precipitate.
p.t. — pneumatic trough.
qcm. — square centimeters.
re-agt. — re-agent.
rec. — receiver.
res. — residue.
r.f. — reducing flame.
r.s. — ring stand.
sat. — saturate.
sep. — separate.
sl. — slight or slightly.
sol. — solution or soluble.
sp. gr. — specific gravity.
s.r. — stirring rod.
Tr. — tartrate.
t.t. — test tube.
up. disp. — upward displacement.
v. — *vide*, see.
vol. — volume.
yel. — yellow.

INDEX TO EXPERIMENTS.

1. Metric Measurements.
2. Glass Manipulation.
3. " "
4. Physical Changes.
5. " "
6. Chemical "
7. Elements and Compounds.
8. Oxygen, Preparation.
9. " Combustion.
10. "
11. Nitrogen, Separation.
12. " and Air.
13. Hydrogen from Acids.
14. " " Water.
15. Carbon, Preparation.
16. " Properties.
17. Combustion.
18. "
19. Positive and Negative Elements.
20. Acids, Bases, Salts.
21. Acid, Alkaline, Neutral Reactions.
22. Soluble Salts by Neutralization.
23. " " from Metals.
24. " " " Carbonates, etc.
25. Insoluble Salts by Precipitation.
26. Hydrochloric Acid.
27. " "
28. Hydrofluoric "
29. Nitric "
30. Aqua Regia.
31. Sulfuric Acid.
32. Ammonium Hydroxid.
33. Fixed Alkalies.
34. Oxids of Nitrogen.
35. Oxids of Nitrogen.
36. Carbon Dioxid.
37. Chlorin.
38. Euchlorin.
39. Bromin.
40. Iodin.
41. Sulfur.
42. Hydrogen Sulfid.
43. Phosphorus.
44. Arsenic.
45. Compounds of Boron.
46. " " Silicon.
47. Oxidation and Reduction.
48. Lead.
49. Silver.
50. Mercury.
51. Group I.
52. Mercury.
53. Lead.
54. Bismuth.
55. Cadmium.
56. Copper.
57. Group II, A.
58. Arsenic.
59. Tin.
60. Antimony.
61. Group II, B.
62. Iron.
63. "
64. Aluminum.
65. Chromium.
66. Group III.
67. Cobalt.
68. Nickel.
69. Manganese.
70. Zinc.
71. Group IV.
72. Barium.
73. Strontium.
74. Calcium.
75. Magnesium.
76. Group V.
77. Sodium.
78. Potassium.
79. Ammonium.
80. Group VI.
81. Borates.
82. Nitrates.
83. Nitrites.
84. Chlorates.
85. Carbonates.
86. Sulfids.
87. Sulfites.
88. Thiosulfates.
89. Fluorids.
90. Chlorids.
91. Bromids.
92. Iodids.
93. Acetates.
94. Cyanids.
95. Tartrates.
96. Oxalates.
97. Silicates.
98. Ferrocyanids.
99. Ferricyanids.
100. Sulfates.
101. Chromates.
102. Phosphates.


BOSTON ENGLISH HIGH SCHOOL
LABORATORY.
DESK
TABLE AND LOCKERS
INSTRUCTOR'S ROOM
CASE
TABLE AND LOCKERS
PLATFORM
APPARATUS CASE
DARK ROOM FOR PHOTOGRAPHY
SINK
TABLE AND LOCKERS
HOOD
DOWN
COAT-ROOM
a. work-tables.
b. elevator.
c. water-baths.
d. shelves.
e. distilled water.
f. blast-lamp.
g. shelf (see details).
h. sliding black-boards.
LECTURE-ROOM BENEATH LABORATORY
61.
WORK-TABLE AND LOCKERS
DOOR OF LOCKER
3 ft. to floor
Barton – 1895.

1. METRIC MEASUREMENTS.

I. Length.

Note the length of 10^{cm} on a metric ruler, then estimate by the eye alone this length on the cover of a book, and on various objects, verifying each result till you can carry in mind 10^{cm}. Estimate the number of inches it covers, and verify. In the same way experiment with 1^{cm}. Measure the perpendicular distance between the blue lines on a sheet of foolscap paper. Measure the diameter of the older nickel five-cent piece. Make a drawing of the following measurements on the opposite page: one square decimeter, *i.e.* 10^{cm} on a side; 1^{qcm}.

II. Volume.

Measure in a graduate 10^{cc} of water, then pour it into a t.t. Note, without marking, what proportion of the t.t. is filled. Make a drawing of it on the opposite page. Pour out the water, then pour into the t.t. as near the same volume as possible, estimating by the eye alone. Verify the result by pouring the water into the graduate. Repeat this till you can estimate quite accurately. Try it with a t.t. of another size. Estimate 1^{cc} of the liquid in the same way. Draw a figure of a cube 1^{cm} on a side.

In future experiments *estimate volumes, without measuring,* unless special accuracy is required.

III. Weight.

Balance a piece of paper on each pan of a pair of scales. On one pan put a 10^{g} weight and balance this with fine salt or sugar. Note with the eye the quantity of the substance, then remove it. Now estimate a like quantity and verify by weighing it. Repeat the experiment several times. Weigh 1 or 2^{g} and estimate as before. See if 1^{g} can be piled on a one-cent coin. Weigh one of the old nickel five-cent pieces. In subsequent experiments *estimate quantity of solids,* unless accuracy is desired.

Supplementary. — Commit to memory the metric tables for length, volume and weight (v. Physics). How many cc in a liter? How many grams should a liter of water weigh? 10^{cc} of water? Hg weighs 13.6 times as much as water. What will $5\frac{1}{2}^{cc}$ of Hg weigh? A rectangular block of marble is $3 \times 4 \times 2^{cm}$ and has sp. gr. 2.5. What does it weigh in grams?

2. GLASS MANIPULATION.

I. Ignition Tubes.

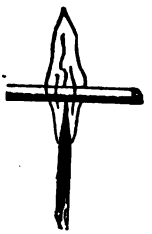


Fig. 1.

Take a piece of ignition tubing 20 or 25^{cm} long, hold it *steadily* in a Bunsen flame *just above the inner greenish cone* (Fig. 1), and rotate it. When it begins to soften draw it out slightly (Fig. 2, *a*) and raise it a little in the flame. Do not pull or twist the glass but let the heat gradually separate it, so as not to have long threads attached. The ends may be rounded (Fig. 2, *b*) by holding in the edge of the flame (Fig. 3). Finally anneal the glass by closing the lower openings of the lamp and holding the heated end in the flame to cover it with soot, then letting it cool gradually without touching any object. Unless great care is taken in this, the glass will break when next put into the flame. Save the tubes for subsequent experiments.

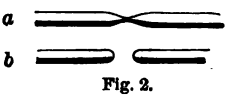


Fig. 2.

II. Stirring Rod.

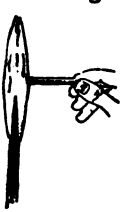


Fig. 3.

Weld the ends of a piece of glass tubing about 15^{cm} long and $\frac{1}{4}$ or $\frac{1}{2}^{cm}$ in diameter by holding each end successively in a Bunsen flame above the green cone (Fig. 3), and rotating till the openings are sealed. Use care in cooling as before.

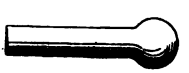


Fig. 4.

Supplementary. — Bulb Tubes. Draw out a piece of tubing as in 1, then melt the closed ends by holding and rotating as in Fig. 3. When the glass is sufficiently softened, blow moderately into the open end, repeating the operation till the tube assumes the desired shape (Fig. 4).

III. Connecting Tubes.

Use an ordinary gas flame (or a fish-tail attachment to a Bunsen burner) for bending glass, as the Bunsen flame is too hot. Turn down the gas till the flame is about 5^{cm} across, then bend each of 3 pieces of tubing, 16^{cm} long, in the middle and at right angles, by letting the glass rest lightly in the fingers of both hands and holding it *lengthwise* of the flame and just above the dark part (Fig. 5). Rotate it *slowly* and hold *very steadily*. When it begins to soften take it quickly from the flame and — holding it between the eye and a window frame — bend it at right angles.

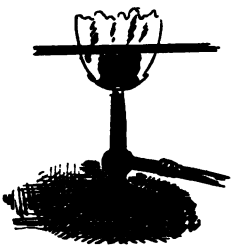


Fig. 5.

If it does not bend easily, heat it again. Bend also one piece of tubing (50^{cm} long), 5 or 6^{cm} from one end. Round the sharp corners of each by holding the tip ends of the tubes in the edge of a *Bunsen* flame for a minute (Fig. 3). Do not let any part of the heated glass touch any object and avoid wiping off the soot till the glass is cool enough to handle, when it should be cleaned.

IV. Delivery Tubes.

Examine the angles (all obtuse) and the relative lengths of the parts in Fig. 6 and in a model, noting that the bends *a* and *c* are very near the ends, and *a* and *b* are close together. Do this work very carefully. Use a piece of glass tubing 50^{cm} long, holding it as before. Make the bends in the order *a*, *b*, *c*. Hold the glass constantly in the flame, turning it occasionally to heat both sides. When it yields remove and bend it *out of the flame*. To make the bends at *b* and *c*, turn the glass quickly and — closing one eye — sight along the tube so as to have all parts lie in the same plane. Round the sharp edges 1 and 2 in the flame, as before, and cool with care. Show all results to the instructor.

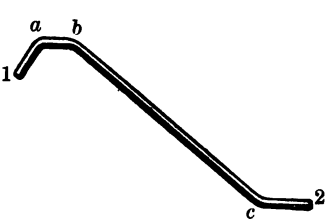


Fig. 6.

V. Perforating Corks.

Fit a cork to a t.t., remove it and with a round file bore a hole in it — holding the cork in the left hand and pressing the end of the file against its *larger surface*. *Observe constantly during the operation whether the perforation is perpendicular to the surface of the cork. The orifice must be perfectly circular and a little smaller than the tube it is to receive.* When completed, adjust to the d.t. and t.t. Detach the stopper from the t.t., not from the d.t.

Solution.

See whether crystals of granulated sugar, $C_{12}(H_2O)_{11}$, can be crushed. Put into a t.t. 5^g or more of the sugar, just cover it with water, H_2O, and boil in a Bunsen flame for a minute, using a t.t. holder of wood, metal, or paper (Fig. 7), till the sugar dissolves. Cool it by holding the tube under a jet of water, and when cool taste a drop of the liquid. Have the sugar particles been divided by dissolving? How do you know? Arrange a filter paper (Fig. 8) and filter the sol., using a filter stand and collecting the filtrate in another t.t. (Fig. 9). Touch a drop of the filtrate to the tongue with a stirring rod (s.r.). Note whether the sugar has gone through the filter paper. Divide the liquid into two parts. Save one for Exp. 6, and dilute the other with an equal vol. of H_2O; then cover the t.t. with the thumb or hand, invert it (Fig. 10) and, *holding it away from the table,* shake the contents vigorously. Taste a drop of the sol. Note whether the sugar particles have been further divided.

Fig. 7.

Fig. 8.

Fig. 9.

Supplementary. — Read very carefully Rule 14 and the Model for Taking Notes. (a) Dis. a little $CuCl_2$ or $Cu(NO_3)_2$ in water, boiling as before, and filter but do not taste the sol. (b) Dis. also a little NaCl, filter and taste the filtrate. Evaporate. Do substances in sol. pass through a filter paper? Many substances like SiO_2 and $BaSO_4$ are not sol. in water or acids, others, like Zn, Cu, etc., are sol. in acids only. (c) Heat a platinum wire in a Bunsen flame. Note if it undergoes change.

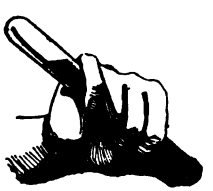


Fig. 10.

Terms: Physical division, physical solution, insoluble, dilution, molecule, evaporation, properties, filtrate, residue.

5. PHYSICAL CHANGES.

Solution and Crystallization.

1. Fill an e.d. ⅓ full of water, heat it, and add small pieces of alum until the liquid becomes thick. Replace the water lost by evaporation. Now put in a narrow piece of cotton cloth so that it lies in the liquid but does not touch the bottom of the dish, and set the latter aside to cool. Observe occasionally, meantime doing the following:

2. Cautiously boil about 5^g copper sulfate in 5^{cc} H_2O in a t.t. until it is dissolved. Then place the t.t. in an oblique position and let the liquid cool. If necessary, leave till the next laboratory hour. Note the formation of crystals in 1 and 2, and observe their form, color, luster, angles, etc. Are these illustrations of physical or of chemical changes, and why? Trace each step in the process. Which seems more soluble, alum or sulfate of copper? Are they more soluble in hot or in cold water?

3. Put a few crystals of copper sulfate into an i.t. or small t.t. and slowly heat. Observe the sides of the tube and any change of color, luster, hardness, and form in the crystals. Explain. When the tube is cool add a drop of water.

4. Experiment in the same way with some of the crystals of alum, tasting the powder obtained by heating.

5. Put a crystal of sodium sulfate and a small piece of calcium chlorid on separate pieces of paper and leave them in the locker exposed to the air till the next exercise. Describe the result with explanation.

Supplementary. — (a) Put into an e.d. as much powdered S as will stay on the point of a very small knife blade. Cover it with a very little CS_2 (keep the flame at a distance from this substance) so as to dis. the S, let it evaporate in a draft of air, then watch closely as the CS_2 evaporates. Describe and explain the phenomena. Put a drop of this sol. on a microscopic slide and watch it closely under the microscope as the CS_2 evaporates. (b) Dissolve and re-crystallize Na_2SO_4. (c) Mix sols. of alum and $CuSO_4$ and try to crystallize the two substances together. Are the above salts soluble or insoluble?

Terms: Crystal, amorphous substance, mother liquor, saturation, supersaturation, water of crystallization, deliquescent, efflorescent, luster.

6. CHEMICAL CHANGES.

I. Sulfuric Acid and Sugar.

To the sol. saved from Exp. 4 slowly add sulfuric acid, H_2SO_4, in a small stream, holding the tube with a t.t. holder, and the bottle as in Fig. 11 (read Rule 9), till the substance changes color. Add a little more acid, and if the sugar solution is strong enough it will cause the substance to swell and run over the tube. Observe the color, odor, and temperature, but do not taste it. Does it appear to be a solid or a liquid? Sugar is a compound of C and H_2O. H_2SO_4 has great affinity for water. From these facts explain the phenomena. Clean the tube with water, using a brush if necessary.

Fig. 11.

II. Heat and Sugar.

See whether heat will break up sugar, by dipping a short piece of glass tubing moistened with water into a little sugar and holding it in a Bunsen flame. Describe all you see, note the odor, and state your conclusions. What produced the chemical change?

III. Combustion of Wood.

Hold a small stick in a flame. State whether the changes observed are chemical, or physical, with reasons.

IV. Changes in Solutions.

Add a little hydrogen chlorid, HCl, to 5^{cc} $Pb(NO_3)_2$ sol. The hydrogen chlorid and the lead nitrate have become lead chlorid and hydrogen nitrate. The former product is insoluble and is therefore thrown down (precipitated). To 5^{cc} of $CuCl_2$ sol. add a little NH_4OH — ammonium hydroxid — and notice any evidence of change.

Supplementary. — (a) To 5^{cc} of $HgCl_2$ sol. add a few drops of KI sol. (b) Add a few drops of KI sol. to 5^{cc} of $Pb(NO_3)_2$ sol. (c) On a few bits of Cu in an e.d. pour a few drops of nitric acid, HNO_3. Describe all you see. Are these chemical, or physical changes? Why?

Terms: Combustion, chemical solution, affinity, precipitation, chemical change.

Synthesis.

Fill an i.t. (from Exp. 2) not over ⅓ full of an *intimate mixture* of one part by weight of S (finely powdered brimstone, or flowers of S) with two parts of fine Cu filings. Observe that the two elements are entirely distinct, and note the color and luster of each. Now heat the tube very slowly for a minute (moving it alternately out of the flame and into it, so as not to break the glass) then heat very strongly, the upper part first (Fig. 12). Watch the contents of the tube for evidence of a chemical change. What is the evidence? When this change has taken place throughout the whole mass let the tube cool for a minute, then heat an instant, break the tube with a jet of water, and examine the substance. Note whether it resembles Cu or S in color or luster. Describe fully what you saw, state what is supposed to have taken place, and give an equation to express it.

Fig. 12.

Supplementary. — (a) Mix Fe filings and S (in about the same proportion as Cu and S), try to separate the Fe with a magnet and the S with CS_2 from a part, then heat the other part in an i.t. Test the product with magnet and CS_2; also by adding a little H_2O and then HCl in an e.d. An offensive gas is liberated which either element alone would not give if similarly treated. Describe fully with equations. (b) Light a match, and as it burns watch the colors of the flame and see if there is any evidence of a product being formed. The match contains the elements P, S, and C, which combine with the element O in the air to form compounds called oxids of P, S, and C (v. Exp. 9). (c) Burn a piece of charcoal and observe. The products are for the most part invisible gases. (d) Heat in an i.t. a little granular pyrite, FeS_2, and finally test the residue with a magnet.

Terms: Synthesis, sulfid, oxid, binary compound, element, gas, luster, residue.

8. PREPARATION OF OXYGEN.

Fill 4 or 5 wide-mouthed bottles — recs. — with water, invert them on the shelf of a p.t. in which the water is 3 or 4^{cm} above the shelf. Mix on a paper 5 or 10^{g} $KClO_3$ (not pulverized) and 3 or 4^{g} of powdered MnO_2. Put the mixture into a large dry t.t. which should not be over $\frac{1}{3}$ full. Adjust a stopper and d.t. and hang the app. on a r.s. (Fig. 13). Direct a Bunsen flame against the t.t., holding the lamp and slowly moving it. Avoid heating too long in one place (take it away from the tube occasionally for a few seconds), or the tube will melt. Watch the bubbles in the d.t., collect the escaping gas in the recs., and notice the bubbles as they burst, and whether the gas has color. Explain, and after a time observe again. Is there evidence of a gas, or a solid, or both? *As soon as the flame is taken away remove the d.t. from the trough* to prevent the water drawing back and breaking the tube. Why is there this back action? Let the recs. remain on the shelf till wanted, or, if taken out, keep them covered with glass plates (Fig. 14). After doing Exp. 9 clean the t.t. by covering the residue with water, closing the mouth of the tube with the thumb or hand, and shaking the contents vigorously, *holding it away from the table.*

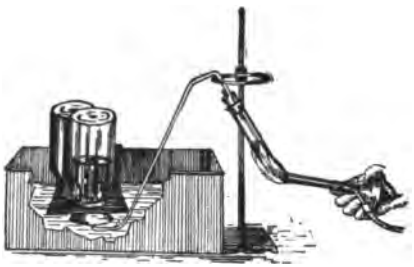


Fig. 13.

Fig. 14.

Supplementary. — Does the O occupy more, or less, volume than the factors from which it was obtained? Why? Would it weigh more, or less? Why? The MnO_2 takes no part in the reaction, but is used to distribute and equalize the heat and lower the temperature of dissociation. Name all the substances in the t.t. at the end of the exp.

Terms: Factors, products, dissociation.

9. COMBUSTION IN OXYGEN.

1. Put a burning splinter into a rec. of O for an instant, note the color and vigor of the flame, then remove, blow out the flame and put in the glowing stick. Repeat till there is no effect. State accurately everything you saw and heard. Wood consists largely of C. Explain the combustion and write an equation for it. Test the product by adding 4 or 5^{cc} of lime water, $Ca(OH)_2$ sol.; shake it well and notice the effect.

2. Make a d.s. by hollowing out the end of a crayon, or electric light carbon, and attaching a Cu wire. Put into it a bit of S, hold the whole in a flame till the S burns, then lower it into a rec. of O (Fig. 15). Notice the color and vigor of the flame, and the color and state of the product, explain what you see and write the equation. Name the product and test it by cautiously noting the odor, and also by putting a drop of $K_2Cr_2O_7$ sol. on paper and holding it in the gas, noting any change of color.

Fig. 15.

3. Put into an e.d. with a pair of forceps (don't handle it) a piece of P, half as large as a pea. Dry it by rolling it lightly on paper, then put it into a d.s. Heat for an instant the end of a wire or file, touch the P with it and lower the spoon at once into a rec. of O. Observe the color of the flame and of the product. Describe, also write the equations. When combustion stops burn every bit of the P by holding the d.s. in a flame. Add a few cc of H_2O to the rec., shake it well and drop in a piece of blue litmus paper.

Supplementary. — What became of the O in the above cases? Would the product in each case weigh more or less than the C, S, P? How do you know? (a) Cover the bottom of a rec. of O with sand or H_2O 1 or 2^{cm} deep. With forceps hold for an instant in a flame one end of a picture-cord wire 5 or 10^{cm} long, then dip it into a bit of powdered S. Hold it again in the flame till the S burns, then thrust it into the rec. of O. If the Fe does not burn try it again, or repeat with another rec. (have but little S on the wire). Describe the combustion and the product, and give the equation. (b) With forceps hold in the flame a piece of Mg ribbon till it begins to burn, then instantly thrust it into a rec. of O. (c) Try Zn tipped with S.

Terms: Supporter of combustion, combustible, kindling point.

10. OXYGEN.

I. Oxygen from Oxids.

Heat strongly 1 or 2^g of HgO in a t.t. or i.t. and thrust a glowing splinter into the tube while still holding it in the flame (Fig. 16). Note the color of the HgO while it is being heated, observe the sides of the tube, and finally pour out and carefully examine the contents. Explain, and give the reaction. Save the residue.

Fig. 16.

II. Oxygen from Nitrates.

Put into a t.t. about 1^g of Pb $(NO_3)_2$, heat it and test the gas as before. Note any snapping, the color of the fumes, which are NO_2 and O (the O being colorless), and the color of the residue, PbO. Also write the equation.

III. Oxygen from Chlorates.

Try to obtain oxygen in the same way by heating a few crystals of $KClO_3$. Test as before, and write full notes.

Supplementary. — What 3 classes of substances have you found which yield O? From which is the oxygen obtained most easily? N_2O will also support combustion of C, S, P, but not as readily as pure O. Heat and test: (a) Pb_3O_4; (b) $NaNO_3$.

Terms: Oxid, chlorate, chlorid, nitrate, residue.

11. SEPARATION OF NITROGEN.

Fill a p.t. with water 3 or 4^{cc} above the shelf. Prepare a d.s. with a wire bent sharply 5 or 6^{cm} from the crayon. Pass the wire through the orifice in the shelf so that it will reach half way up an inverted rec., fasten it there (so as to be easily removable) with the mouth of the rec. under water (Fig. 17). When adjusted remove the rec., leaving the wire. Using forceps and e.d. put a piece of P (the size of half a pea) into the spoon. Touch it with a warm (but not too hot) wire or file handle, and instantly invert the rec. over it as before, *pressing the latter down* and holding it steadily with the hand till combustion ceases. Was there any escape of gas? Before removing the d.s. be sure the combustion has stopped, and let no air enter. Compare the combustion and the product with the same in the oxygen exp. Describe fully with equation. Finally remove the crayon without admitting air or disturbing the rec. Burn any remaining P in a flame and leave the rec. till the gas becomes tolerably clear, then remove with a glass plate, turning the rec. right side up and *keeping the water in the bottom.* Set this aside for Exp. 12.

Fig. 17.

Supplementary. — (a) Try to separate the N in air with S in a d.s., setting the S on fire by holding a flame over the adjusted spoon. (b) Try paper or charcoal. What elements do these exps. show to exist in the air? N may also be separated by forcing air over red hot Cu, which forms CuO. It may be obtained from compounds, such as nitrates and ammonia.

12. NITROGEN AND AIR.

I. Properties of N.

(1) Put a burning stick into the rec. of N (Exp. 11), sliding along the glass cover enough to admit it. Note the effect on the stick and on the gas. Try it with a glowing stick. Is the spark rekindled, or extinguished? (2) See whether the P, S, and C on the end of a match will burn in the gas. Is there any equation? Why? Is N a combustible? A supporter of combustion? (3) Test the liquid with blue litmus paper. Describe and explain.

II. Composition of Air.

Measure accurately the water in the rec. of N by pouring it into a graduate and recording the volume in cc. Then measure the total capacity of the rec. in the same way, and *compute the percentage of O and of N* (by volume) in the air, giving the computation and remembering that the first vol. of water represents the vol. of O burned. If any air was forced out in Exp. 11, will the per cent of O found be too large or too small? Explain.

Supplementary. — Compare the properties of N with those of O. Air contains also CO_2 (tested for by Exp. 36) and H_2O (shown by setting a pitcher of cold water in a warm room). Make tests for each and explain. From the percentage of N there must be subtracted about 1% of the recently discovered element argon, A, a gas more inert than N.

Terms: Inert substance, percentage by volume and by weight, acid, litmus paper.

13. HYDROGEN FROM ACIDS.

I. Preparation.

Fill 4 or 5 recs. with water and invert in a p.t., then put into the gen. (Fig. 18) (or use the same app. as for making O) 8 or 10^{g} granulated Zn, cover with H_2O and add 5^{cc} of HCl through the thistle tube. Have the bearings perfectly tight, and, without applying heat, collect the gas like O. Observe and describe the action and note any heat. If action stops add more HCl. Leave the recs. of gas in the p.t. till wanted in II (or if removed keep them inverted, Fig. 19). Has the gas any color? Why? Clean the app. and save the Zn.

Fig. 18.

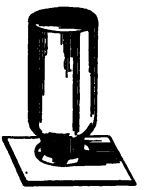


Fig. 19.

II. Tests.

(1) Lift a small rec. of H, hold it inverted, and thrust a burning stick into the gas. Note any explosion and whether the gas or splinter burns. (2) Try it with another rec. and then cautiously look into the open mouth of the latter, noting the colors of flame. Explain fully the explosion, combustion, etc., with equation. Note the (3) color and (4) odor of (the impure) H. (5) Collect by up. disp. a t.t. of H, and test by putting it mouth to mouth with a t.t. of air and then bringing the two open tubes successively to a flame.

Supplementary. — (a) Put 2 or 3 small pieces of Zn into a t.t. (use a t.t. holder), cover it with water and add a little H_2SO_4. Notice the action, test the combustibility of the gas, and write the equations. (b) Put into a t.t. a few Fe filings, add H_2O and HCl. Test the gas as before. (c) Try Fe and H_2SO_4. Make a complete record of all work. From what class of substances has H been obtained, and how? Is H a combustible? A supporter? (d) Try the action of Zn and H_2SO_4, without H_2O, cautiously noting the odor. (e) Pass H into euchlorin (Exp. 38). (f) Fill a rec. half full of water, invert it in a p.t., letting air fill the upper half. Now fill the rest with H from a gen., then bring the mouth of the rec. to a flame. (g) Make a philosopher's lamp (Fig. 20), but before lighting the hydrogen, test the gas by collecting a t.t. full by upward displacement and bringing it to a flame. If a sharp sound ensues there is danger, as the gen. contains air. If only a low muffled sound, the gas may be lighted. Lower a larger open glass tube over the flame. Other flames give the same result.

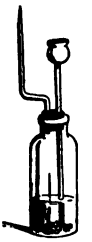


Fig. 20.

Terms: Upward and downward displacement, philosopher's lamp, singing flame.

14. HYDROGEN FROM WATER.

I. Action of Na on H_2O.

(1) Put into a clean rec. 15 or 20^{cc} of H_2O. Have a cover of paper or cardboard, not glass. Observe the color and luster of freshly cut Na; take a small piece from the naphtha in which it is kept (using forceps and *dry* e.d.), drop it into the rec., and cover the latter at once (Fig. 21). Describe the action, note any fumes or gas (try to ignite it), and write equations. When action stops take off the cover and bring the fumes near a Bunsen flame, or hold the lower openings of the burner close to the mouth of the rec. and notice the color imparted to the flame. This is the flame test for Na and its cpds. Are the fumes solid or gaseous, and what becomes of them?

Fig. 21.

(2) Hold a piece of moist red litmus paper in the fumes, also put one into the liquid. Test the liquid by adding a little to these sols.: (3) $FeCl_3$; (4) $FeSO_4$; (5) $AlCl_3$.

(6) Try the action of Na on hot H_2O by boiling a little H_2O in a t.t. and pouring it into a rec., then dropping a piece of Na on it and covering it at once. Note all the phenomena and write equations for: (a) the action of Na on H_2O; (b) the combustion of H, and (c) Na; (d) the sol. of Na_2O.

II. Action of K on H_2O.

Perform the exp. as above, using K and cold H_2O, and making similar observations, notes, and tests, including flame tests.

Supplementary. — Which has greater affinity for H_2O, Na or K? (a) Collect H by putting a small piece of Na in a wire cage and quickly thrust it under an inverted t.t. full of H_2O in a p.t. Test the gas. What proportion of H in water is replaced by K or Na? Are the metals heavier or lighter than H_2O? (a) Moisten a piece of paper, put it into a rec. and drop on the moist paper a bit of Na and cover it. When action stops try to light the fumes by bringing a Bunsen flame to the mouth of the rec. (b) Try the same with K.

16. PROPERTIES OF CHARCOAL.

I. Carbon a Reducing Agent.

Fill an i.t. not over ¼ full of an *intimate mixture* of CuO and C (powdered charcoal), about 7 parts CuO to 1 part C by weight. Heat it very cautiously at first, then strongly till you see evidence of a chemical change. What is the evidence? What caused the mixture to rise? After the action stops is there any change in color? When sure that no more action will ensue, cool it for a minute and pour the contents into a dry e.d. Examine it carefully as to color and luster. Explain, giving an equation to express the chemical change.

Finally test the residue by adding 2 or 3^{cc} HNO_3, noting the color of the fumes and of the residue. See whether HNO_3 has a similar effect on the original mixture of CuO and C. What does the test show?

II. Carbon a Decolorizer.

Shake up 5 or 10^{g} of bone black — animal charcoal — in a t.t. or rec. with 10 or 20^{cc} cochineal sol., then filter and notice the color of the liquid. If not perfectly colorless pour it again on the same filter, using more carbon if needed. Try in the same way sols. of indigo and litmus; also of $K_2Cr_2O_7$ and $KMnO_4$. Can you draw any general inference of the decolorizing action on animal, vegetable, and mineral pigments, the first three being animal or vegetable, the last two mineral? $K_2Cr_2O_7$ may be turned slightly yellow by alkali in the bone black.

III. Carbon a Deodorizer or Disinfectant.

Prepare a sol. of H_2S (Exp. 42). Notice the odor of the gas and its solubility. Put into a rec. 5 or 10^{g} of powdered charcoal and pour upon it 10 or 20^{cc} H_2S sol. and shake the mixture well. Then pour the whole on a filter, collect the filtrate in a clean t.t. and see if any odor remains; if so, mix and filter again, using more charcoal if necessary. The H_2S has been absorbed physically into the pores of the coal and partially oxidized there. This illustrates the absorbent action of charcoal on gases, animal effluvia, and microorganisms in the sick room.

17. EXPERIMENTS IN COMBUSTION.

I. Structure of Flame.

(1) Make a careful examination of the parts and colors of a candle flame, and make a drawing to show them. Move the candle slightly in the air to show the outer flame. This is best seen in a dark room. Describe fully. (2) Examine the structure of a Bunsen burner (unscrew the top if necessary), make a drawing to show the orifices, and state what use they subserve. Replace the tube and light the gas. Hold the flame in front of a dark object (as a blackboard), examine the parts, make a drawing, give a brief description, and state the color of each part. If possible, put the flame in direct sunlight, and study the parts from its shadow, to confirm your results.

II. Combustion of Flame.

(1) Put a moist stick across the base of the Bunsen flame for an instant, and notice what parts are burned. Hold a stick just above the inner cone of the flame. Make sketches of results. (2) Press quickly down on the flame with a paper, or pasteboard, remove before it burns, and notice the shape of the charred part, or press down on the flame with a fine wire gauze, and observe by the glowing of the wire where the heat is most intense. Sketch.

(3) Put one end of a small d.t. into the inner blue cone, not into the lamp, and try to light the gas at the other end. (4) Thrust a match quickly into the inner cone near the bottom. If done quickly the phosphorus will not take fire. The match contains a supporter and a combustible. Why does it not burn? In what parts of the flame does combustion occur?

Supplementary. — Examine: (a) an ordinary gas flame; (b) the flame of a kerosene lamp; (c) the flame of a burning splinter, or match, holding it vertical and breaking off the charred part as fast as it forms. Have all flames practically the same structure?

III. Light of Flame.

(1) Sprinkle a very little charcoal dust into a Bunsen flame, and note any change of light or color, or, better, dip a fine splinter into powdered charcoal and thrust it into the openings at the base. Notice flame. (2) File a nail or other metallic substance over the flame, or sprinkle in CuO, or "iron by hydrogen." (3) Also stir up the dust near the flame, or tap the lamp with the fingers. What produces flame? What is the cause of light in a flame, and what flames give light? (4) Close the orifices at the base of the burner, and explain the change of light. (5) Set fire to a small piece of charcoal, by holding it in a flame, and see whether it burns with flame or only glows. What does this show?

IV. Kindling Point.

(1) Light the gas and hold a fine wire gauze 3 or 4^{cm} above the burner. Why does it not burn above the wire? (2) Extinguish, then relight the gas above the gauze. Result and explanation. Gradually lift the wire till the gas will not burn. (3) Again light the gas above the gauze, and hold another gauze above the flame, so as to confine it above and below. State three conditions of combustion, define each, and show how this exp. illustrates them.

Supplementary. — Why does a lamp smoke? Why does phosphorus take fire spontaneously? Why does gunpowder explode? Why will it burn without air? Why do we exclude the air to put out a fire? Why does blowing a flame sometimes cause it to burn, and sometimes extinguish it? Why does cannel coal burn with flame? When will a mixture of H and air not explode? Why will some substances burn in water? See which has the lower kindling point, P or S, by putting each on one end of a piece of iron at equal distances from a flame which heats the other end.

Terms: Kindling temperature, spontaneous combustion, combustible, supporter.

I. Dis. Ag in HNO_3 and ppt. Ag with Cu.

Put a ten-cent silver coin into an e.d. and add 10 or 20 drops of HNO_3. Warm it, if necessary, over asbestos, and watch the result as to action, color of liquid, color of fumes, etc., and give the reason for each, with equation. When action stops, or the coin is dissolved, add 10^{cc} H_2O, then pour it into a t.t. and put in a copper wire (Fig. 23). Leave for 10 to 30 minutes. At this point begin II., but observe any deposit on the wire, noting its color, luster, texture, crystalline form, and any change in the color of the liquid. Explain, giving equation. Save the Ag. Which has greater affinity for NO_3, H, or Ag? Why is H not set free?

Fig. 23.

II. Dis. Cu in HNO_3 and ppt. Cu with Pb.

Put into a t.t. or e.d. a thin piece of sheet Cu, say 1^{qcm}; add 10 or 20 drops HNO_3. Make as accurate observations and notes as before, including equation. After dissolving the Cu add 10^{cc} H_2O, and put in a Pb wire (or other piece of Pb), made bright with a file or by just dipping it into a little HNO_3. Leave it for some time in the Cu $(NO_3)_2$ sol., and begin III. Afterwards notice the deposit, the color of liquid, etc., as before, and take full notes with equations. Which has more affinity for NO_3, Cu, or Ag?

III. Dis. Pb in HNO_3 and ppt. Pb. with Zn.

Dis. part of a small piece of Pb in a few drops of HNO_3, heating if necessary, and when action ceases add 10^{cc} H_2O; pour the liquid into a t.t. and put in a piece of Zn. There should be scarcely any free acid when the Zn is introduced, or violent effervescent action will ensue and both Pb and Zn will dissolve. Make full notes as before, including equations.

Supplementary. — (a) Obtain crystals of pure $AgNO_3$ from a coin. (b) Of $Pb(NO_3)_2$ from Pb. (c) Of $Cu(NO_3)_2$ from Cu. (d) Make $CuSO_4$ from Cu with dil. acid. (e) See whether Zn will ppt. Cu from $CuSO_4$. (f) From $CuCl_2$. With 1^{gs} of pure Ag, how much $AgNO_3$ could be made? How much Ag will 5^{g} of Cu replace from a sol. of $AgNO_3$?

Terms: Chemical solution, physical solution, precipitation, stochiometry.

20. ACIDS, BASES, SALTS.

I. Acids.

(1) Pour into an e.d. a few drops of HCl, add 4 or 5^{cc} H_2O, stir it, taste a drop from the s.r. (Fig. 24). Dip the end of a strip of blue litmus paper into it. State results. (2) Rinse the e.d., then put in a few drops HNO_3 and 5^{cc} H_2O, stir and test as before. (3) Test in like manner H_2SO_4. What do these exps. teach you of acids?

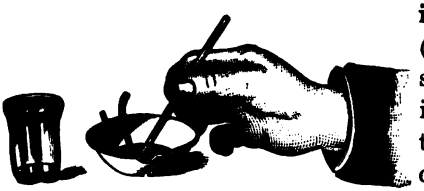


Fig. 24.

II. Bases.

(1) Pour into an e.d. 1 or 2^{cc} NH_4OH, add 4 or 5^{cc} H_2O, stir it, and taste a drop. If it has no taste add more NH_4OH. Dip the end of a piece of red litmus paper into it. (To redden blue litmus hold it in the *fumes* of HCl. To turn red litmus paper blue hold it in NH_3 *fumes*.) (2) Rinse the e.d. and test $NaOH$ sol. in the same way. Also (3) KOH sol. What do these exps. teach concerning bases?

III. Salts.

(1) Pour into a clean e.d. 1 or 2^{cc} of $NaCl$ sol., taste a drop of it, and test it with litmus, red and blue. Test in the same way (2) KNO_3 sol., also (3) a sol. of $(NH_4)_2SO_4$. What is the usual action of salts on litmus? Test, however, these sols. with both colors: (4) Na_2CO_3; (5) $CuSO_4$; (6) $HNaCO_3$; (7) $HNaSO_4$.

Supplementary. — Test sols. of: (a) H_3PO_4; (b) $H_2C_2O_4$; (c) $HC_2H_3O_2$; (d) $Ca(OH)_2$; (e) $Ba(OH)_2$; (f) HNa_2PO_4; (g) $Na_2B_4O_7$; (h) NH_4NO_3.

Terms: Acid, base, salt, acid salt, normal salt, alkaline salt, neutral salt, salt acid to litmus, alkali, fixed alkali, volatile alkali, alkaline carbonate.

21. ACID, ALKALINE, AND NEUTRAL REACTIONS.

Put into a small beaker or a t.t. 5^{cc} of an aqueous sol. of blue litmus. (1) Pour a few drops of HCl into an e.d., beaker, or t.t. and dip a s.r. into this (never dip a rod into a reagent bottle), then stir the litmus solution with it, noting any change of color. If none occurs add another drop of acid with the rod. (2) Pour a few drops of NH_4OH into a clean e.d. or t.t., dip the s.r. into it, then stir the litmus solution with this. If the color is unchanged repeat till blue is again obtained. (3) In the same way put a drop (or more, if necessary) of HNO_3 from an e.d. into the same blue solution. (4) When it is reddened add a drop or more of NaOH sol. until it becomes blue. (5) Change it again to red with a drop of H_2SO_4, and (6) restore the color with KOH sol.

Supplementary. — (a) Add a few drops of H_2O to a sol. of red litmus, and also to one of blue. (b) Add to both red and blue litmus sols. a sol. of NaCl. (c) Try NH_4Cl sol. (d) KNO_3 sol. (e) $HNaCO_3$ sol. (f) $HKSO_4$. (g) $CuSO_4$.

Terms: Acid, alkaline, and neutral reactions.

22. SOLUBLE SALTS BY NEUTRALIZATION.

I. Sodium Chlorid.

Pour into an e.d. not over 5^{cc} NaOH sol., and into a clean t.t. or beaker 4 or 5^{cc} HCl. Now add some of this latter, a little at a time (Fig. 25), to the NaOH sol., stirring it meantime till the product is neutral to litmus paper; when nearly neutral add a drop at a time with a s.r. and test with both colors, using only the end of the paper and keeping it on the side of the e.d. If blue litmus paper is reddened by the sol., add NaOH sol., a few drops at a time. If red litmus is turned blue add a drop of HCl. When the sol. does not affect either red or blue litmus left in it for a minute, show it with both papers in it to the instructor, then filter and evaporate the liquid to dryness by boiling it over a plate and abestos paper (Fig. 26). When it is cool, examine the residue and taste it. What is the object of evaporation? Explain what took place when the acid and base were mixed, and give the equation to express it. Sift a little of the salt into the flame. The color shows a cpd. of Na. Test for Cl by Exp. 27.

Fig. 25.

Fig. 26.

II. Potassium Sulfate.

Into an e.d. put 5^{cc} KOH sol. and neutralize it with dilute H_2SO_4, using both test papers. When sure it is neutral, evaporate most of the water and taste the product. Explain the action, giving the equation. Sift a little into a flame. The color shows a K cpd. (best seen through blue glass). Test SO_4 by Exp. 31.

III. Ammonium Nitrate.

Neutralize NH_4OH with HNO_3 (preferably dilute) in the same way as before. Stop evaporation when dense fumes begin to appear. These fumes indicate a decomposition of the salt by heat (v. Exp. 34).

Supplementary. — How would you make by neutralization $NaNO_3$, Na_2SO_4, KNO_3, KCl, NH_4Cl, $(NH_4)_2SO_4$? Write also equation for each. Weak acids will not neutralize strong bases, and *vice versa*. Hence, salts like Na_2CO_3 are not neutral. How do these exps. illustrate the law of definite proportion?

Terms: Neutralization, double decomposition, definite proportion, evaporation.

22. SOLUBLE SALTS BY NEUTRALIZATION.

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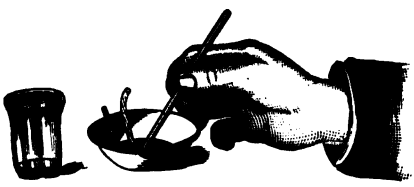

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Into an e.d. put 5cc KOH sol. and neutralize it with dilute H_2SO_4, using both test papers. When sure it is neutral, evaporate most of the water and taste the product. Explain the action, giving the equation. Sift a little into a flame. The color shows a K cpd. (best seen through blue glass). Test SO_4 by Exp. 31.

III. Ammonium Nitrate.

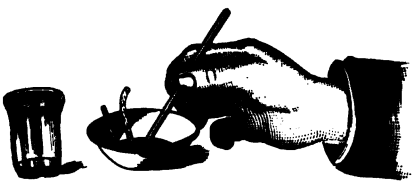
Neutralize NH_4OH with HNO_3 (preferably dilute) in the same way as before. Stop evaporation when dense fumes begin to appear. These fumes indicate a decomposition of the salt by heat (v. Exp. 34).

Supplementary. — How would you make by neutralization $NaNO_3$, Na_2SO_4, KNO_3, KCl, NH_4Cl, $(NH_4)_2SO_4$? Write also equation for each. Weak acids will not neutralize strong bases, and *vice versa*. Hence, salts like Na_2CO_3 are not neutral. How do these exps. illustrate the law of definite proportion?

Terms: Neutralization, double decomposition, definite proportion, evaporation, fumes.

I. Sodium Chlorid.

Pour into an e.d. not over 5^{cc} NaOH sol., and into a clean t.t. or beaker 4 or 5^{cc} HCl. Now add some of this latter, a little at a time (Fig. 25), to the NaOH sol., stirring it meantime till the product is neutral to litmus paper; when nearly neutral add a drop at a time with a s.r. and test with both colors, using only the end of the paper and keeping it on the side of the e.d. If blue litmus paper is reddened by the sol., add NaOH sol., a few drops at a time. If red litmus is turned blue add a drop of HCl. When the sol. does not affect either red or blue litmus left in it for a minute, show it with both papers in it to the instructor, then filter and evaporate the liquid to dryness by boiling it over a plate and abestos paper (Fig. 26). When it is cool, examine the residue and taste it. What is the object of evaporation? Explain what took place when the acid and base were mixed, and give the equation to express it. Sift a little of the salt into the flame. The color shows a cpd. of Na. Test for Cl by Exp. 27.



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II. Potassium Sulfate.

Into an e.d. put 5^{cc} KOH sol. and neutralize it with dilute H_2SO_4, using both test papers. When sure it is neutral, evaporate most of the water and taste the product. Explain the action, giving the equation. Sift a little into a flame. The color shows a K cpd. (best seen through blue glass). Test SO_4 by Exp. 31.

III. Ammonium Nitrate.

Neutralize NH_4OH with HNO_3 (preferably dilute) in the same way as before. Stop evaporation when dense fumes begin to appear. These fumes indicate a decomposition of the salt by heat (v. Exp. 34).

Supplementary. — How would you make by neutralization $NaNO_3$, Na_2SO_4, KNO_3, KCl, NH_4Cl, $(NH_4)_2SO_4$? Write also equation for each. Weak acids will not neutralize strong bases, and *vice versa*. Hence, salts like Na_2CO_3 are not neutral. How do these exps. illustrate the law of definite proportion?

Terms: Neutralization, double decomposition, definite proportion, evaporation, fumes.

23. SOLUBLE SALTS FROM METALS.

I. Zinc Chlorid.

To a piece of Zn in a t.t. add water and a little HCl. Describe and explain the action, and when it ceases, pour the liquid into an e.d. and evaporate to dryness. Observe and taste the salt. Name it and write the equation. To test the salt, add a little H_2O to dissolve it and divide the sol. into 4 parts. Test for Cl by Exp. 27, and for Zn by Exp. 70.

II. Iron Sulfate.

Dissolve as above a few Fe filings in dil. H_2SO_4. When action stops add H_2O, filter, and evaporate the filtrate. Then dissolve the salt in water after having examined it. Name it and write the equation. Test it for Fe by Exp. 63, and for SO_4 by Exp. 31.

III. Copper Nitrate.

Dissolve a little Cu in dil. HNO_3, pour off the liquid, evaporate, and then add H_2O. Test for Cu by Exp. 56, and for NO_3 by 82. Make full notes.

Supplementary. — The salts in Exps. 22, 23 are sol. in water. Insoluble salts are not made in this way. $PbCl_2$ is only partially insoluble, hence HCl will not dissolve much Pb. Table A shows that there are but two soluble salts of Pb; hence most acids do not act upon that metal. By reference to this table you can judge what acids will probably act on a given metal. Will H_2SO_4 dis. Ba? Pb? Cu? Will HCl probably dis. Sn? Ag? Hg? Mg? Will H_3PO_4 dis. Pb? Ca? Ag? (a) Try any of the above. (b) Try to dis. Pb in $HC_2H_3O_2$. Compare chemical sol., as shown in 23, with physical sol., Exp. 4. What two processes in chemical sol.?

Terms: Insoluble, soluble, saturation, supersaturation.

24. SOLUBLE SALTS FROM CARBONATES, HYDROXIDS, OXIDS.

I. Carbonates.

(1) To a piece of $CaCO_3$ (marble or limestone) in a t.t. add H_2O, and a little HCl. When the action stops, pour a little of the liquid into an e.d., evaporate and notice the residue. See if it will dis. in H_2O. State your observations and the reaction. Test for the presence of Ca by Exp. 74. (2) Try Na_2CO_3 (or $HNaCO_3$) with HCl. (3) $CaCO_3$ may be tried with H_2SO_4 or HNO_3.

II. Hydroxids.

(1) To some $Fe(OH)_3$ (made by adding NH_4OH to $FeCl_3$ sol., and pouring off the supernatant liquid after the ppt. settles) add drop by drop just enough HCl to dissolve it. (2) Try also HNO_3 and H_2SO_4 on separate ppts. of $Fe(OH)_3$. Evaporate and dis. in H_2O as before. Test for Fe by Exp. 63. (3) Try $Al(OH)_3$ (made from $AlCl_3$ sol. and NH_4OH) with HCl. Evaporate, dis. in H_2O, and test for Al by Exp. 64.

III. Oxids.

To 2[g] PbO (litharge) in an e.d. add some dil. HNO_3 and heat it. Decant on a filter, partly evap. the filtrate, add H_2O, and test for Pb by Exp. 48.

Supplementary. — Notice from Table A what carbonates and hydroxids are soluble. (a) Add H_2O to a little Na_2CO_3 (or K_2CO_3) in a t.t. and boil a minute. (b) In another tube cautiously add HCl to Na_2CO_3. Explain the difference between (a) and (b). (c) Into (a) pour cautiously a little HCl. Describe the action and explain it with reaction. Sol. salts may be made from many other classes of cpds. besides the above.

Terms: Physical and chemical solution, decantation.

25. INSOLUBLE SALTS BY PRECIPITATION.

Insol. Salts of Pb.

(1) Pour into a t.t. 5^{cc} $Pb(NO_3)_2$ sol. and add a few drops HCl. (2) Try it again, substituting NaCl sol. for HCl. (3) Use also a sol. of NH_4Cl. Record results and write equations. (4) Now in place of $Pb(NO_3)_2$ use a sol. of $Pb(C_2H_3O_2)_2$, try the action of the same three reagents as previously used, and make a similar record. What is the ppt. in each case? Could you obtain the same ppt. by putting together any other sols., and if so, what ones?

To 5^{cc} of each of the following sols. in a t.t. add a little sol. of $Pb(NO_3)_2$ or $Pb(C_2H_3O_2)_2$ and write the equation, underlining the ppt. and writing the color above. (5) K_2CrO_4. (6) KI. (7) KBr. (8) NaOH. (9) Na_2CO_3. (10) Na_2SO_4. (11) K_4FeCy_6.

Supplementary. — What are the only two soluble salts of Pb? Law of Ppn.: *Whenever a mixture is made of two sols., one of which contains the positive part and the other the negative part of an insoluble cpd., a ppt. of the insol. cpd. will always form.*

From Table A what salts of Ba are insol.? (a) Try to make some of them by the application of the above law. (b) Also try $Fe(OH)_3$. (c) Make HgS. (d) Try to make MnS. (e) Make $CaCO_3$.

Terms: Insoluble, precipitation, positive, negative.

I. Sodium Chlorid.

Pour into an e.d. not over 5cc NaOH sol., and into a clean t.t. or beaker 4 or 5cc HCl. Now add some of this latter, a little at a time (Fig. 25), to the NaOH sol., stirring it meantime till the product is neutral to litmus paper; when nearly neutral add a drop at a time with a s.r. and test with both colors, using only the end of the paper and keeping it on the side of the e.d. If blue litmus paper is reddened by the sol., add NaOH sol., a few drops at a time. If red litmus is turned blue add a drop of HCl. When the sol. does not affect either red or blue litmus left in it for a minute, show it with both papers in it to the instructor, then filter and evaporate the liquid to dryness by boiling it over a plate and abestos paper (Fig. 26). When it is cool, examine the residue and taste it. What is the object of evaporation? Explain what took place when the acid and base were mixed, and give the equation to express it. Sift a little of the salt into the flame. The color shows a cpd. of Na. Test for Cl by Exp. 27.

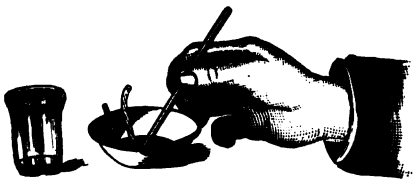

Fig. 25.

Fig. 26.

II. Potassium Sulfate.

Into an e.d. put 5cc KOH sol. and neutralize it with dilute H_2SO_4, using both test papers. When sure it is neutral, evaporate most of the water and taste the product. Explain the action, giving the equation. Sift a little into a flame. The color shows a K cpd. (best seen through blue glass). Test SO_4 by Exp. 31.

III. Ammonium Nitrate.

Neutralize NH_4OH with HNO_3 (preferably dilute) in the same way as before. Stop evaporation when dense fumes begin to appear. These fumes indicate a decomposition of the salt by heat (v. Exp. 34).

Supplementary. — How would you make by neutralization $NaNO_3$, Na_2SO_4, KNO_3, KCl, NH_4Cl, $(NH_4)_2SO_4$? Write also equation for each. Weak acids will not neutralize strong bases, and *vice versa*. Hence, salts like Na_2CO_3 are not neutral. How do these exps. illustrate the law of definite proportion?

Terms: Neutralization, double decomposition, definite proportion, evaporation, fumes.

27. HYDROCHLORIC ACID, HCl.

Properties and Tests.

(1) Test the liquid in each bottle (Exp. 26) with blue litmus paper. (2) Taste a drop, using a s.r. (3) Cautiously note the odor. (4) Put a piece of Zn into a t.t. and pour over it 5 or 10^{cc} of the contents of the first Wolff bottle. (5) If a gas escapes, test its combustibility. Explain fully with observations and equations. (6) Put into a t.t. 5^{cc} $Pb(NO_3)_2$ sol. and add a little of the liquid from the first bottle. If a ppt. falls, pour a small part of it into another t.t., add a little H_2O, and boil a minute. Does it dissolve? What does that show? Now add a little $K_2Cr_2O_7$ (or K_2CrO_4) sol. Explain. (7) To 5^{cc} $AgNO_3$ sol. add a little of the acid. If a ppt. falls let it settle, then pour off most of the supernatant liquid, leaving the residue; then add NH_4OH and shake it. Finally acidify cautiously with HNO_3. Explain all results. (8) Test $HgNO_3$ sol. in the same way, adding NH_4OH at the end, but not HNO_3. By any of the above exps. can you determine which bottle contains the stronger acid?

Supplementary. — (a) Add HCl to $Cu(NO_3)_2$ sol. and explain the result. (b) Separate Ag from Cu in a mixture of solutions of their nitrates. Try the action of HCl on sols. of: (c) $Co(NO_3)_2$; (d) $NaNO_3$; (e) KNO_3; (f) $MgSO_4$. What 3 chlorids are insol. in H_2O, and how are they made?

Terms: Analysis, reagent, pungent, acidify, alkalize.

28. ETCHING WITH HYDROFLUORIC ACID, HF.

Cover thinly with beeswax or paraffin one side of a small piece of glass. Spread the wax evenly over the surface, by warming the other side of the glass and pouring off the excess of melted wax. With a sharp metallic point, as the handle of a file, mark some design through the wax when cold (Fig. 28). Put into a lead dish (it will spoil a porcelain e.d.) a very little HF sol. (be exceedingly careful not to get any on the flesh) and with a camel's-hair brush spread some of it over the design on the glass. Do this several times. Finally, after a few minutes wash away the acid from the glass, melt and pour off the wax, wiping off what remains with a cloth wet in naphtha, or alcohol. Examine closely the result and describe it. By reason of its affinity for SiO_2, HF acts on the glass and forms SiF_4. The equation should be written.

Fig. 28.

Supplementary. — Etch a t.t. so as to make a graduate of it. Why cannot HF sol. be kept in glass bottles? HF is made by treating powdered CaF_2 (fluorite) with H_2SO_4 in a lead dish. Write the equation (v. Exp. 89 for other tests). The fumes must not be inhaled.

Terms: Etching, fluor spar, fluorid, fluorite, silica.

29. NITRIC ACID, HNO_3.

I. Preparation.

Introduce into a glass retort through the tubulure, *holding the former so as not to have the chemicals touch the neck of the retort* (Fig. 29), 10 or 15^g fine $NaNO_3$ (or KNO_3) and 20 or 25^{cc} H_2SO_4.

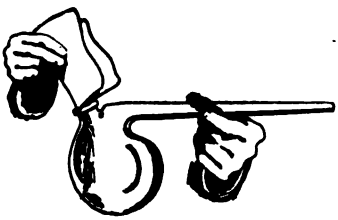

Fig. 29.

Adjust the apparatus as in Fig. 30, having the neck of the retort pass to the bottom of a wide t.t., which is surrounded by water in a wide tumbler, or bottle. The water is to cool and condense the acid from the vaporous state in which it passes over. Apply the Bunsen flame, using a plate and asbestos, for 15 or 20 minutes. Notice the liquid running down the neck of the retort, also any colored fumes — name and account for the latter, and see whether they increase with the heat — the color of the liquid in the t.t., and the cause of it. Do the ordinary reactions (which should be written) account for the fumes? Observe also the color of the product in the retort. Heat may have reduced some of the white Na_2SO_4 to dark-colored Na_2S. Use great care not to get any of the acid on the hands. The antidote, after thorough washing, is $HNaCO_3$ (v. Rule 19).

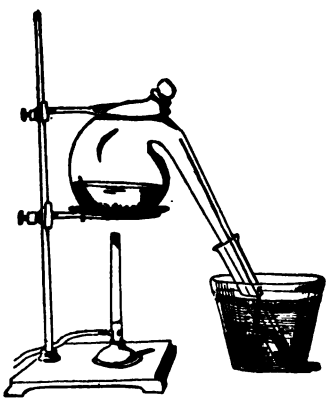

Fig. 30.

II. Tests.

(1) Test the vapor from the end of the retort with a few drops of NH_4OH. (2) Put a drop or more of the acid on a *very few* Cu filings in an e.d. Observe and explain with equation. (3) Try with a s.r. the action of a drop on the finger-nail, then wash it off and add a drop of NH_4OH. This shows its action on animal matter. (4) Try it also on a feather or on silk. (5) Try its decolorizing action on 3 or 4^{cc} of indigo sol. in a t.t. Try its action on insoluble salts, e.g.: (6) CuS; (7) $CaCO_3$; (8) $BiOCl$. State what this shows of the solubility of the respective nitrates (v. also Exp. 82).

Supplementary. — (a) Compare the action of HNO_3 and H_2SO_4 on wood. Could HNO_3 be made from $Pb(NO_3)_2$? How are nitrates made? (b) Make $Zn(NO_3)_2$. (c) Try to dis. a little Sn in HNO_3 in an e.d. (d) Try Sb. (e) Put a drop of commercial HNO_3 on writing paper, another on newspaper. The latter contains wood pulp. (f) Try to decolorize anilin solutions.

Terms: Fuming nitric acid, metastannic acid, xanthoproteic acid, salt of nitric acid, nitrate, tubulure.

30. AQUA REGIA.

I. Preparation.

Pour 2 or 3^{cc} of HNO_3 into a t.t. containing 6 or 8^{cc} of HCl. If there is no evidence of chemical action, warm the mixture a minute and observe. To what is the color due? Give reaction.

II. Tests.

(1) Into a part of the aqua regia introduce a little gold leaf by winding it around a moist s.r., not letting it touch the sides of the t.t. (2) Into another part drop a bit of Pt foil, $\frac{1}{2}^{qcm}$. If no action ensues, heat it. Pt does not dis. as readily as Au. Try the solvent action of aqua regia on: (3) Zn; (4) Fe; (5) Cu; (6) Pb. Give the usual explanations, reactions, etc., for each of the above exps. Why should not the acid act as well on Pb as on the others?

Supplementary. — See if Au will dis. in: (a) HCl, or (b) HNO_3. Also try (c) Pt. Try Hg in (d) HCl, then in (e) aqua regia. (f) Try Fe in each and notice the color of each liquid after filtering. Explain. (g) Test the 2 chlorids of Fe by Exp. 63.

Terms: Nitro-hydrochloric acid, gold chlorid, ferrous and ferric chlorids.

31. SULFURIC ACID, H_2SO_4.

I. Preparation.

Burn a small piece of S in a rec. in which there is H_2O enough to cover the bottom, using a d.s. and confining the fumes as much as possible by covering the mouth of the rec.

Pour into an e.d. 1 or 2^{cc} HNO_3, and dip into this a piece of strong paper, or cloth, or a wood shaving, and lower it into the rec. of SO_2, holding it there for some time without dropping any acid from it into the rec. See whether the fumes change color. Repeat the operation several times. Explain the phenomena and give all reactions. Cover the rec. with the hand and shake the liquid and the gases well together.

II. Tests.

(1) Apply the litmus test. (2) Put a drop of the liquid on writing paper, and evaporate it so as not to burn the paper. See whether the paper is charred, blackened, or made rotten by the liquid. Explain. This is a test for free H_2SO_4. (3) Add a little of the liquid to 5 or 10^{cc} $BaCl_2$ sol. (or $Ba(NO_3)_2$) in a t.t., state the effect and give an equation. If you obtain a ppt., add a little HCl and shake it well. Does it dissolve? (4) Pour into a t.t. containing 5^{cc} H_2O the same amount of H_2SO_4 from the shelf bottles. Observe any thermal effects on this mixture and explain. Save the dilute acid in a separate rec. (5) Apply tests (2) and (3) above to this mixture. (6) Pour a *few drops* of concentrated acid upon 1 or 2^g sugar in an e.d. (7) Dip a small stick into a t.t. which contains 1 or 2^{cc} H_2SO_4. (8) Pulverize a bit of starch, add a few drops of the acid. If no change occurs, cautiously heat it for a minute. Sugar is $C_{12}(H_2O)_{11}$; starch is $C_6(H_2O)_5$; cellulose is $C_{18}(H_2O)_{15}$; explain the action of the acid.

Supplementary. — Why is commercial H_2SO_4 liable to be of a brown color? (a) Measure accurately 10^{cc} of concentrated acid in a graduate, leave it in an open t.t. or rec. for a week, then measure it carefully again. Explain the result. (b) Test commercial acid for $PbSO_4$ by pouring a little into an equal vol. of H_2O in a t.t. $BaSO_4$ is the only salt of Ba that is not dis. by HCl. (c) Test Na_2SO_4 sol. with $BaCl_2$. Explain. (d) Test a sol. of Na_2SO_3 for Na_2SO_4.

Terms: Salt of sulfuric acid, fuming sulfuric acid, sulfate, starch, cellulose.

32. AMMONIUM HYDROXID, NH_4OH; AMMONIA, NH_3.

I. Preparation.

Powder and put into a flask about 10^g NH_4Cl and 7^g $Ca(OH)_2$ (freshly slaked is best). Add 20^{cc} H_2O and connect with Wolff bottles containing H_2O (Fig. 31), as in the HCl exp. Heat 10 or 15 minutes, using a plate and asbestos paper. Observe the phenomena, especially the pressure in the different bottles and the absorption of gas, and write the reaction. While the mixture is heating, do III.

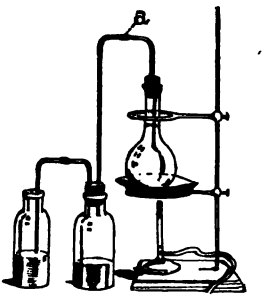

Fig. 31.

II. Tests.

(1) Disconnect the flask while the gas is escaping (*a*, Fig. 31), put 2 or 3 drops HCl into an e.d., and pass the gas over it from the gen. Observe the fumes, giving equation. (2) Let the flask cool as it stands, and test the liquid in each bottle (odor and litmus). Put sols. (5^{cc} each) of the following substances into separate tubes, and add a little of the prepared liquid to each: (3) $FeSO_4$; (4) $FeCl_3$; (5) $AlCl_3$. Observe colors, especially of ferrous and ferric cpds. (6) Test likewise $CuCl_2$ sol., first with very little NH_4OH, then with excess. State and explain all phenomena.

III. Ammonia.

Mix 2 or 3^g of fine NH_4NO_3 with an equal quantity of $Ca(OH)_2$, put them into a t.t., and warm it over a flame. See whether a gas escapes by: (1) cautiously taking the odor; (2) testing with red litmus paper; (3) with HCl.

Supplementary. — (a) Put into a t.t. a gram or two of either $(NH_4)_2SO_4$, NH_4NO_3, NH_4Cl, or $(NH_4)_2C_2O_4$, and add a little NaOH (or KOH) sol., warm it, and apply tests. What two classes of cpds. are used to obtain ammonia? Are the above examples of double decomposition, and why? Relation between ammonia and ammonium hydroxid? (b) Fill a t.t. with NH_3 by upward displacement, and invert it over a rec. of water. Observe and explain.

Terms: Volatile alkali, fixed alkali.

33. FIXED ALKALIES.

I. Sodium Hydroxid, NaOH.

To 5^{cc} of a saturated sol. of Na_2CO_3 add about an equal quantity of $Ca(OH)_2$ sol. Shake the mixture well and observe. If a ppt. falls, name it and write the equation. What goes into solution? Let the ppt. settle, then add some of the supernatant liquid to sols. of: (a) $Fe(SO_4)$; (b) $FeCl_3$; (c) $AlCl_3$. Effects, colors, and equations should be given in each case. If any effect different from what is expected occurs, it should be noted and explained.

II. Potassium Hydroxid, KOH.

Treat a sat. sol. of K_2CO_3 in precisely the same way as Na_2CO_3 above, making close observations and taking full notes as before, including all the tests given.

Supplementary. — Compare the action of fixed alkalies with that of NH_4OH on sols. of Fe and Al salts. (a) Obtain some NaOH or KOH in the solid state. In what other ways were NaOH and KOH made in the laboratory? (b) Would $Ca(OH)_2$ act on NaCl to form NaOH? (c) $Ca(OH)_2$ on Na_2SO_4? (d) Is $FeCl_3$ sol. acid, alkaline, or neutral? (e) Is $Al(OH)_3$ soluble in excess of NaOH or KOH sol.?

Terms: Hydroxid, hydrate, alkali metals, metals of alkaline earths.

34. OXIDS OF NITROGEN.

I. Nitrogen Monoxid, N_2O (Nitrous Oxid).

(1) Put into a flask (of 200^{cc} capacity) 10^{g} of NH_4NO_3, not too coarsely crystalline (so as not to break the flask in melting). Connect the flask with a large t.t. (resting in a rec. of H_2O, Fig. 32), or with a rec. *which contains no water* (Fig. 33), and from this t.t. or rec. have a d.t. leading to a p.t., so as to collect the gas over water. Have the joints tight and the tubes pass through the stoppers. Apply heat not too rapidly, notice the order of changes and give the main reaction. Are there any fumes in the flask? Is the gas colored, and why? Obtain 2 recs. of gas, then remove the lamp and take the d.t. from the water, letting the flask cool on the r.s. (2) Taste *a drop* of the liquid in the large t.t. or rec., and also test with litmus, red and blue. Some NH_4NO_3 will probably have been driven over, also some NO, NO_2, HNO_3, etc., formed by breaking up of NH_4NO_3. (3) Test the gas with a burning stick, also with a glowing one. (4) Test the combustibility of S (well burning when introduced), and (5) of P. Compare the brilliancy of flame and the products obtained with those in O, and write all equations.

Fig. 32.

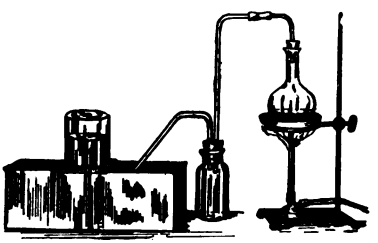

Fig. 33.

Supplementary. — Compare the action of heat on NH_4NO_3 with that on $Pb(NO_3)_2$ in Exp. 10. Compare with the action of $NaNO_3 + H_2SO_4$ (Exp. 29). Learn the symbols and names of all the oxids of N. Try the action of heat (using a t.t.) on: (a) KNO_3; (b) $Hg(NO_3)_2$; (c) $Zn(NO_3)_2$; (d) $Pb(NO_3)_2$.

Terms: Laughing gas, nitrous oxid, nitrates, nitrites.

II. Nitrogen Dioxid NO or N_2O_2 (nitric oxid).

(1) Arrange the app., with inverted recs., as for the hydrogen exp. Then put into the gen. (or t.t.) 5^g Cu turnings, cover them with water and add through the thistle tube 5^{cc} of HNO_3. Collect 2 or 3 recs. of the gas. If action stops add a little HNO_3 (or Cu, if necessary). Save any Cu that is left. Explain the different colors of the gases and the color of the liquid. (2) Test the gas with a burning stick. Explain the fumes. Test also with (3) S and (4) P (well burning). In (4) see whether the solid gradually separates from the gas. Take full notes.

III. Nitrogen Trioxid N_2O_3 and Tetroxid NO_2 or N_2O_4 (peroxid).

(1) Lift a rec. of the gas NO, just prepared, from the shelf of the p.t. What is the effect, what is the new gas, and how is it formed? (2) Heat 1 or 2 crystals of $Pb(NO_3)_2$ in a t.t., and while the gas escapes insert a glowing stick into the tube. (3) Put a fragment of starch into a t.t. and add a few drops of HNO_3. Heat cautiously.

Supplementary. — State how the oxids of N are derived from HNO_3 and nitrates. What action have heat and reducing agents on nitrates? (a) Notice the action of direct sunlight on strong HNO_3 in stoppered bottles. (b) Try the action of heat on other nitrates, *e.g.*, $Cu(NO_3)_2$, KNO_3. (c) Heat a bit of powdered charcoal on a plate or asbestos, and when it is hot add a drop of HNO_3 with a s.r. N_2O_3 and NO_2 are both red and not distinguishable. Find how N_2O_5 is prepared, and its relation to HNO_3.

Terms: Nitrous and nitric anhydrids, nitrogen peroxid, nitric oxid.

36. CARBON DIOXID, CO_2.

I. Preparation.

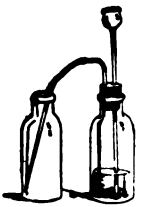

Fig. 34.

Put 10 or 15^g $CaCO_3$ (marble, in lumps) into a t.t. or gen. (Fig. 34), cover with H_2O and — after having the app. all prepared — add 5^{cc} HCl. Add more of the reagents as needed. Enough must be used to produce vigorous action. Attach a delivery tube and collect a rec. of gas by downward displacement, having the tube reach to the bottom of the rec. It will take only a minute.

II. Tests.

(1) Test the gas with a burning stick. (2) Let the gas bubble from a d.t. into 5^{cc} $Ca(OH)_2$ sol. in another t.t. and look for a ppt. Let the action continue until the liquid clears. (3) When the sol. is clear, boil it for a minute and notice the reappearance of the ppt. Explain all the phenomena very fully, giving equations at the proper places. (4) Pass the gas into a sol. of blue litmus. (5) Observe the Ca flame (bring the open mouth of the CO_2 gen. close to a Bunsen flame). Small particles of $CaCl_2$ are carried up by the gas. (6) Also dip a stick into the sol., hold it in the flame and notice the color. (7) Take 2 or 3^{cc} $Ca(OH)_2$ sol. in a clean t.t. and breathe into it through a glass tube. What does the result show? (8) Put a little lime water into an e.d. and look for a scum after a few minutes; or draw the air through a rec. of lime water (Fig. 35), by suction, applying the lips at *b*. The air enters at *a*, and any CO_2 is drawn through the lime water. Explain.

Fig. 35.

Supplementary. — (a) See whether you can obtain CO_2 from Na_2CO_3. Use an open t.t. and test the gas with a drop of $Ca(OH)_2$ sol. on a s.r. (b) Use also $HNaCO_3$. (c) K_2CO_3. (d) Test some oyster, clam, or other shells with a drop of HCl, and see whether there is effervescence; or put them into a gen. and test the gas. (e) Obtain $CaCl_2$ from the CO_2 gen. (f) Test that salt for Cl (Exp. 90). (g) For Ca (Exp. 74). (h) Obtain $CaSO_4$ from $CaCO_3$. (i) $Ca(NO_3)_2$.

Terms: Carbonate of lime, quicklime, slaked lime, lime water, milk of lime, sulfate of lime.

I. Preparation from Hydrochloric Acid.

Put into a flask (or t.t.) 5^g MnO_2 (preferably granulated) and 10^{cc} HCl. Shake them well together, apply gentle heat, and avoid inhaling the gas, but collect it by downward displacement in a rec. loosely covered with paper. Add more HCl if it is needed. Observe and describe, with equations. Note when the rec. is filled, to avoid passing the gas into the room. If accidentally any is inhaled, the antidote is vapor of alcohol (inhale from a handkerchief); or NH_3 will do. State the color of the gas, its sp. gr. compared with air, its effect on the nasal organs.

II. Tests and Properties.

(1) Drop into the rec. a piece of dry Turkey-red cloth (or of calico), also bits of printed and written paper. If there is little or no effect add a very little water, shake the gas, water, cloth and paper well together, and note the effect. Which bleach better, wet or dry fabrics? (2) Into another rec. of Cl (dried if possible) file a little Sb (or stibnite, Sb_2S_3). Describe the result and give an equation. Is this a case of ordinary combustion? Pass some Cl from the gen. into sols. of: (3) $AgNO_3$; (4) $Pb(NO_3)_2$; (5) H_2S.

Supplementary. — (a) Put into a small beaker 5^g bleaching powder, $CaCl_2O$, set this into a larger rec. or beaker, putting into this last the substance to be bleached. Cover the large rec. with pasteboard, through which passes a thistle-tube into the smaller. Pour through the thistle-tube 5^{cc} dilute H_2SO_4 (half H_2O and half acid). Add more, if needed. (b) Mix a gram of fine NaCl with a gram of MnO_2, add in a t.t. 2^{cc} H_2O and 3^{cc} H_2SO_4, and heat. Observe any gas. Test it with NH_3, or otherwise. (c) Dip some unglazed paper into oil of turpentine. $C_{10}H_{16}$, in an e.d.; warm it by holding the paper near a flame for an instant (or heat the turpentine in an e.d. over a waterbath), but do not set it on fire. Then at once thrust the paper into a rec. of Cl. Explain the combustion and describe all you saw, giving the equation. Clean the rec. with naphtha or petroleum.

Terms: Bleaching, bleaching powder, calcium hypochlorite, chlorid of lime.

38. EUCHLORIN, ClO_2, Cl.

I. Preparation.

Drop into a t.t. 3 or 4 crystals of $KClO_3$. Add a *few drops* HCl, heat for an instant and then add 5 or 10^{cc} H_2O.

II. Tests.

(1) Observe carefully the action, noting the color and odor (cautiously), and give equation. (2) To 2^{cc} indigo sol. in a t.t. add a little euchlorin. Is the color discharged? Test in the same way sols. of (3) litmus and of (4) cochineal. (5) Try $K_2Cr_2O_7$ sol. (a mineral pigment; the other three are vegetable or animal coloring matters). What do you conclude from this? (6) Very cautiously add 2 or 3 drops of H_2SO_4 to 2 or 3 small crystals of $KClO_3$ in a t.t. at arm's length from the operator, then fill the t.t. with H_2O. Describe the result with equation. (7) Try to bleach a piece of Turkey-red cloth or calico by putting it into some euchlorin and leaving it a few minutes.

Supplementary. — (a) Try to bleach $KMnO_4$ sol. Will euchlorin dis.: (b) Au? (c) Zn? (d) Cu? Try its action on sols. of: (e) $AgNO_3$; (f) $Pb(NO_3)_2$; (g) $HgNO_3$.

Terms: Euchlorin, oxids of chlorin, detonation.

39. BROMIN AND BROMIDS.

I. Preparation of Br.

(1) Make some starch paste by pulverizing a piece of starch not larger than a pea, adding 10^{cc} H_2O and boiling several minutes in a t.t.; cool it and dil. with 10^{cc} H_2O, shaking it well. Boil again if necessary. [Save part of this for subsequent exps.] (2) Mix 1 or 2 crystals of KBr with an equal quantity of MnO_2, put them into a t.t. and add a few drops H_2SO_4. Heat the mixture and observe carefully. Write the equation, and explain the action.

II. Tests.

(1) Dip a strip of paper into the starch paste, wind it around the end of a s.r. and carefully put it into the t.t., so as not to touch the sides. Then heat the mixture in the t.t. Observe the effect of the Br on the starch. The product is supposed to be starch bromid. Avoid getting much Br into the air in the room, or inhaling it. Alcohol vapor, or ammonia, is the antidote, as for Cl. Do you notice any effect of Br vapor on the eyes? (2) To a sol. of KBr add a few drops of euchlorin. Describe and explain, stating which is the more stable or stronger cpd., KBr or KCl, and how this exp. shows it. (3) Add to a few drops of starch paste 10^{cc} H_2O, shake it well, add a few drops of KBr sol. and one drop of euchlorin. Describe and explain.

Supplementary. — Liquid Br should be examined. (a) Try the action of Br on a mixture of starch paste and KI sol. Try the action of H_2SO_4 on (b) a few drops of KBr sol., also on (c) a crystal of KBr. (d) Pour a little Br vapor from a gen. into 5^{cc} of KI sol. (e) Put a drop of CS_2 into a t.t. with a few drops of KBr sol., then add a few drops of euchlorin and shake it. Notice the color of the CS_2 at the bottom of the tube. The Br was set free and dis. by the CS_2.

40. IODIN AND IODIDS.

I. Preparation of I.

Prepare I like Br in the previous exp., using KI instead of KBr. Notice the difference in its action, color, state, etc., and give a full description with equation.

II. Properties and Tests.

(1) Make the same tests with starch as in Exp. 39, noting the different results. (2) Warm 1 or 2 crystals of I in a *dry* t.t. Notice the color of vapor, and its relative weight (pour a little into a dry e.d.). (3) While warming the tube hold a s.r. half way down the t.t. Look for a sublimate on the rod and on the sides of the tube, examining carefully its form, luster, and color. (4) Put into a small t.t. 1 or 2 crystals of I, add 3 or 4^{cc} of alcohol, C_2H_5OH, and warm a minute. Explain, then (5) add a drop of this with a s.r. to a drop of starch sol. shaken up with 10 or 15^{cc} of H_2O. Observe the color of the product (starch iodid), and boil it a moment to see whether the color disappears. If not, too much I was used. When it is cool (hold the tube under a jet of water) observe again.

Supplementary. — (a) See whether a crystal of I will dis. in H_2O. To determine this apply the starch test. (b) To 5 or 10^{cc} H_2O add a drop or two of CS_2 and of KI sol. (examine), then one drop of euchlorin. Shake well, let stand, and note the color of the CS_2 globule. (c) Test the solubility of I in ether $(C_2H_5)_2O$, keeping the latter away from the flame. (d) Obtain crystals by letting the ether sol. evaporate. (e) To 10^{cc} H_2O add a few drops KI sol. and a few drops of starch paste. Shake well together and note the color. Now add *one drop* of euchlorin (Exp. 38). If too much euchlorin is added the sol. becomes green or black (ppt. of I). Describe the coloration and explain fully. (f) Repeat (e) of Exp. 39 with KI sol.

Terms: Sublimation, sublime, crystal, tincture.

41. EXPERIMENTS WITH SULFUR.

I. Crystallization.

Into a small beaker or large t.t. put 10 or 15^g S, and slowly melt it over a lamp (using sand bath or asbestos), not allowing it to become darker than amber-color. When it is melted remove the lamp, leave the beaker in position (or hold the t.t. in the hand), and when crystals begin to shoot across the surface, pour the liquid S remaining into water in an e.d. Explain.

Loosen the S by pouring round the edges a little HNO_3. Warm if necessary, when the mass may be removed with a thin knife-blade.

II. Allotropy.

Put 10^g S into a t.t. and *slowly* melt it. Notice the yellow color, and see that the liquid (now above 100°) is very thin. Slowly continue to heat it till it becomes black. Note that it is now very thick and can scarcely be poured (about 200°). Apply more heat till it grows thin again (above 300°). Now heat to boiling (over 400°), note the color of the vapor, and any sublimate on the t.t. If it takes fire (S burns at 230°), cover the mouth of the tube with paper or cardboard for a minute. Pour the S into an e.d. of water. Pull it and note its elasticity. Is it now crystalline, or amorphous? See whether it afterward changes. Clean the t.t. as before.

III. Solubility.

Place in an e.d. $\frac{1}{2}^g$ pulverized brimstone and cover it with CS_2 (avoid a flame, as CS_2 is explosively inflammable). See whether any dissolves. Let it stand till the CS_2 has evaporated, watching it meantime. Describe fully the phenomena.

Supplementary. — (a) Try to dis. a little S in ether, $(C_2H_5)_2O$, and let it evaporate. Is S soluble in H_2O? (b) Grind together on a brick with a pestle or other implement, not over a half gram of S and $KClO_3$. (c) Make gunpowder by mixing intimately very fine KNO_3, C, and S (a small quantity of each). Pile the mixture on a brick and cautiously set it on fire. If this reaction is correct, in what proportion should the constituents be mixed: $2KNO_3 + 3C + S = K_2S + 3CO_2 + 2N$? (d) Obtain flowers of S by sublimation and see whether they are crystalline or globular (use compound microscope). (e) Add HCl to a sol. of $Na_2S_2O_3$ and look for a S ppt. (f) Obtain SO_2 from S, also from H_2SO_4. (g) Try to bleach with it a green leaf or a blue petal.

Terms: allotropy, allotropic, fusion, crystalline, globular, amorphous.

42. HYDROGEN SULFID.

I. Preparation and Tests.

(1) Put into a gen. (rec. or t.t.) 5^g FeS, 10^{cc} H_2O, and 5^{cc} HCl (or H_2SO_4). Adjust a d.t., having the joints perfectly tight, and pass the gas for a minute or two into 5^{cc} H_2O in another t.t. or beaker (Fig. 36). Note the odor. Can you determine whether the gas is soluble in H_2O? (2) See whether the sol. is acid, alkaline, or neutral. With a s.r. put a drop of H_2S sol. on bright coins of (3) Ag and (4) Cu. State the effect, products, reactions. (5) Put a drop of $Pb(C_2H_3O_2)_2$ sol. on paper and pass the gas over it from the tube of the generator. Describe the effect (this is the characteristic test for H_2S) and write the equation. Is there any other way in which you would recognize this gas? (6) Try the combustibility of H_2S, using a philosopher's lamp. (7) If it burns, hold a glass tube or a crayon in the flame. Explain.

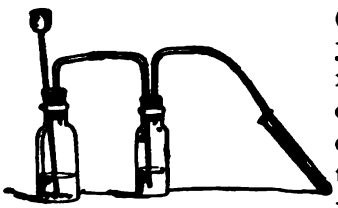

Fig. 36.

II. As a Reagent.

Add a little H_2S sol. to 5^{cc} of each of the following sols., describe the results, write equations where necessary, and explain why there is no effect in certain cases: (1) $HgCl_2$, (2) $CuCl_2$, (3) $BaCl_2$, (4) $CaCl_2$. (5) Next mix 3 or 4^{cc} $CuCl_2$ sol. and the same quantity of $BaCl_2$ sol., add H_2S sol., shake it well and filter. What is the residue and what does the filtrate contain? (6) Now to the filtrate add a sol. of Na_2CO_3. State the effect, give the reaction, and explain how the Cu and the Ba have been separated.

III. Test for Sulfids.

To a little Na_2S or CaS add H_2SO_4. If any gas arises (heat if necessary) try the $Pb(C_2H_3O_2)_2$ test.

Supplementary. — See whether you can separate (a) Cu from Ca; (b) Ca from Hg; (c) Hg from Ba; (d) Fe from Cu. How are some insol. sulfids made? (e) Test FeS for a sulfid? What sulfids are insoluble?

Terms: sulfuretted hydrogen, sugar of lead, sulfid.

43. PHOSPHORUS.

Caution: Phosphorus must be handled with forceps, as the burns from it heal very slowly, and all experiments with it must be done cautiously. None should be dropped upon the floor, nor left upon the apparatus, and all must finally be burned. CS_2 must be kept away from the flame.

I. Spontaneous Combustion.

(1) Into an e.d. put 10 or 20^{cc} of CS_2 (for a class) and drop into it 3 or 4 small pieces of P. Notice the odor of CS_2 and whether the P dissolves in it. Also observe the color and luster of newly cut P. (2) Dip a piece of unglazed paper into the sol., then place it on the ring of a r.s. till it is dry, looking out for any action. If such occurs *state fully what has caused it* and whether it is a case of ordinary combustion.

II. Combustion under Water.

Put into a large t.t. 4 or 5 crystals of $KClO_3$, cover it with H_2O and add a small piece of P. Rest the t.t. in a rec., and then slowly add 2 or 3^{cc} H_2SO_4 through a thistle tube which reaches to the P and $KClO_3$. Account for the phenomena. What color is the liquid, and what is the cause of it? $HKSO_4$, $HClO_4$, ClO_2 and H_2O are formed. Write the equation for forming them, and for the combustion. What compound of P is finally formed?

Supplementary. — (a) Put a small piece of P into a dry e.d. in a dark room, observe, and explain the result. (b) Put a piece of P on a *dry* plate or e.d., set it on fire and at once cover with a dry inverted rec. When the fumes settle add a very little water. Test the product with litmus. (c) Cover a small piece of P with bone black in a plate, and leave for some time. If no action ensues burn the P before leaving the Exp.

Terms: spontaneous combustion, phosphorescence.

I. Marsh's Test.

Caution: AsH_3 is a deadly poison, and must not be inhaled. If the H is ignited before introducing the As compound there is little danger.

Prepare a hydrogen gen., with or without a $CaCl_2$ drying attachment, and with lamp tube (Fig. 37, or model). Put in a few pieces of arsenic-free Zn, some distilled water, and C.P. HCl. After a few minutes test the H gas (Exp. 13) before lighting, and when no sharp explosion occurs light the gas, and observe the color of the flame. Test for As by holding an e.d. over the flame. Now pour through the thistle tube 1 or 2^{cc} of a sol. that may be supposed to contain some cpd. of As. This sol. may be obtained either by dissolving HNa_2AsO_3 in H_2O with a little HCl, or by immersing in HCl a small piece of green paper tag from which any As that may be present will dis. as $AsCl_3$. Note any change in the color of the flame; hold an e.d. or other cold body in the flame and look for a deposit; put a Bunsen flame under the drawn out tube (Fig. 37) and look for a sublimate just beyond. Absence of a sublimate in either case indicates absence of As. Explain fully, writing all equations.

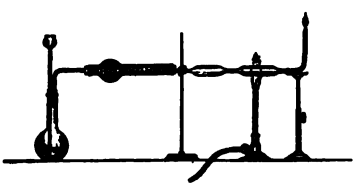

Fig. 37.

II. Reinsch's Test.

Into a sol. of an As salt (obtained by HCl as above) in a beaker or e.d. put a small strip of bright Cu foil and heat for a few minutes. Notice that the foil changes color. After a few minutes remove it, dry it by carefully pressing between folds of blotting paper, cut it into strips, and put them into a t.t.; heat the latter. Notice the vapor and sublimate, As_4O_6, on the tube. This sublimate may be examined under the microscope after first breaking the tube. Explain all results, using equations.

Supplementary. — (a) Reduce As_4O_6 as follows: Mix intimately a bit of it not larger than a wheat grain with as much powdered charcoal, put the mixture into a small t.t. or i.t. and heat strongly. Note the vapor, color, sublimate. (b) Make Scheele's green by mixing sols. of $CuCl_2$ and HNa_2AsO_3 (or other sol. cpds.) and having them nearly neutral. (c) Try the effect on these mixtures of (d) excess of HCl, (e) excess of NH_4OH. (f) See whether As_4O_6 is very soluble in H_2O by putting a *very little* into a t.t. and heating with H_2O. (g) Heat a little powdered arsenopyrite, FeAsS, in a t.t. and note the odor. (h) Test some wall paper, or colored silk, for As.

45. COMPOUNDS OF BORON.

I. Boric Acid.

Put 5^g of $Na_2B_4O_7$ into a t.t., add 10^{cc} of dil. H_2SO_4 and boil till it is clear, adding more water if necessary. Then cool it by holding the tube under a jet of water, keeping watch for crystals. When it is cool pour off the supernatant liquid and rinse the crystals once or twice with water to remove the H_2SO_4, then put them into an e.d. Add a very little water, and when the crystals have dissolved dip into the sol. a piece of turmeric paper, which should then be dried and the color noted. Describe fully the results.

II. Borax Beads.

Make a loop in the end of a Pt wire (which is welded into a short glass tube, Fig. 38). Moisten the loop end and dip it into some powdered borax, $Na_2B_4O_7$. Now heat it, using the mouth blowpipe, dipping it again into the borax if more is wanted, and heating till a clear transparent bead is obtained. Describe and explain the phenomena. Beads may be colored as follows: If blue is wanted dip the clear bead into a very dil. sol. of some Co salt, as $CoCl_2$, and fuse again. (If too much Co is used it will look dark.) For other colors v. Table G. Detach the beads by suddenly jerking the Pt wire while the bead is fused. Save the beads, and label them, stating the coloring material.

Fig. 38.

Supplementary. — (a) See whether H_3BO_3 is more, or less, soluble in hot water than in cold. (b) Make a bead of H_3BO_3 if possible. (c) Stir a little H_3BO_3 in an e.d. with a few drops of C_2H_5OH. Then take some of the mixture on a short glass tube and hold it in a Bunsen flame, noting the color imparted to the latter. (d) Try the same with $Na_2B_4O_7$, (e) then with $Na_2B_4O_7$ to which has been added a few drops of H_2SO_4.

Terms: borax bead, flux.

46. COMPOUNDS OF SILICON.

I. Silicic Acid and Silica.

Pour a few cubic centimeters of water glass, H_4SiO_4, into an e.d. and add a little HCl. Note the phenomena, describe the result and write the equation. Pour off the extra HCl and rinse the residue, H_4SiO_4, 2 or 3 times with H_2O. Now heat it over a Bunsen flame, with stirring. This leaves SiO_2. Write the equation, and note how the product feels when rolled between the fingers. Is silicic acid made differently from other acids? Pulverize (in a mortar if necessary) some of the SiO_2 obtained, to use in the following.

II. Silicates by Solution.

Make a strong sol. of NaOH (or KOH) and add to it some of the SiO_2 obtained above, very finely powdered. Heat and stir the mixture for some time. See whether the SiO_2 dissolves, and write an equation.

III. Silicates by Fusion, Glass.

Make a small mixture of equal parts of powdered Na_2CO_3 and K_2CO_3 and fuse the mixture into a bead as in the case of borax. Note that the bead is opaque when cold. Is it so when hot? Dip this bead when hot into a little SiO_2 powdered very finely, then heat again more strongly in the hottest part of the flame. If it is not transparent when cold, dip it again into the SiO_2 and heat more strongly than before. See whether it now resembles glass. Describe all phenomena and write the equations. Colors may be obtained as in borax beads, Table G.

Supplementary. — (a) Try to dis. finely powdered SiO_2 in a strong sol. of Na_2CO_3, boiling the mixture. (b) Try to fuse SiO_2 in a borax bead.

Terms: fusible, infusible, fusion, transparent, opaque, silica, silicate, glass, water glass.

47. OXIDATION AND REDUCTION.

I. Oxidation.

(1) Into each of two tubes put 5cc $FeSO_4$ sol. (freshly dissolved). To one of these add a few drops HNO_3 and boil a minute, then cool. To each of the tubes add 5cc $NaOH$ sol. (or NH_4OH). Observe the color effects, explain their causes, and give equations for the ppts. Explain the action of HNO_3.

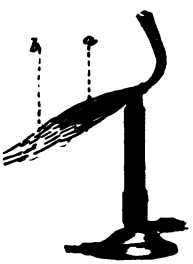

Fig. 39.
a, reducing flame.
b, oxidizing flame.

(2) Put a small fragment of Pb on a piece of charcoal, slightly hollowed out with the handle of the forceps. Fit a metallic tube to a Bunsen burner, and with a mouth blow-pipe blow the *oxidizing flame* (outer) *strongly and steadily* against the Pb for 4 or 5 minutes (Fig. 39). What first takes place? As you stop blowing, notice the yellow vapor that escapes from the globule of Pb: also the yellow coating of PbO on the charcoal. Explain fully, using equations.

II. Reduction.

(1) Pour a little $FeCl_3$ sol. into two tubes. Pass SO_2 gas, or H, from a gen. into one of these for a few minutes. Next add NH_4OH to each. Compare the products and explain, with equations. What action has the SO_2 or the H? Try the action of Zn and H, or of Zn alone.

(2) Put $\frac{1}{2}$g PbO on a piece of charcoal hollowed out as before. With the blow-pipe blow the *reducing flame* (inner) steadily against it for some time, or until a metallic globule is obtained. What is it? Explain fully, with equation. See whether the globule is sectile (can be cut) or malleable (can be flattened by pressure). *Extinguish the fire*, if the coal still glows, *with a drop of water.*

Supplementary. — Notice that the action of the flames here is like that of oxidizing and reducing agents generally. (a) Experiment with other metals and oxids. (b) See whether $Fe(OH)_2$ or $Fe(OH)_3$ tends to change to the other on standing. Which is the more stable cpd.? (c) Try sols. of $FeSO_4$ and $FeCl_3$ in the same way. (d) Try to oxidize $FeSO_4$ sol. with a little euchlorine. (e) Oxidize $HgNO_3$ with HNO_3. (f) Try to reduce $K_2Cr_2O_7$ sol. to $CrCl_3$ with a little HCl, C_2H_5OH and heat. Note the change in color, also test by Exp. 48. Are these oxidizing, or reducing agents: (g) $KClO_3$, (h) KNO_3, (i) C, (j) Cu? (k) Pulverize about a gram of KNO_3 and mix it with a like quantity of fine charcoal. Put into a dry i.t. and heat. (l) Try KNO_3 and S in the same way.

Terms. — Oxidation, reduction, oxidizing agent, reducing agent, oxidizing flame.

48. LEAD, Pb″.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. $PbCl_2$** (v. Ag, Hg′). To $Pb(NO_3)_2$ sol. add HCl, filter (test fil.), wash, reject washing (and fil. if no more $PbCl_2$ falls).	(1) **$PbCl_2$**	white	
2. **Dis. $PbCl_2$ in hot H_2O.** (v. Ag, Hg′). Pour on res. (1) boiling H_2O from t.t. Save fil. Repeat this several times.			(a) **$PbCl_2$**
3. **Final test for Pb.** To first filtrate (a) add $K_2Cr_2O_7$ sol. (or K_2CrO_4).	(2) **$PbCrO_4$**	yellow	

Supplementary. — 1. $PbCl_2$ is somewhat sol. in H_2O, hence traces of it are tested for in Grp. II. 3. If there is much free acid in a sol. the $K_2Cr_2O_7$ test is not to be relied on. H_2SO_4 should then be used. What other Pb salt might be used for sol. (Table A)? (a) Allow some of the hot water sol. of $PbCl_2$ to cool in a t.t. and look for crystals. Explain. (b) See which is the stronger cpd. $PbCl_2$ or $PbCrO_4$. (c) Make several other final tests for Pb.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENT.	REMARKS.
1	**AmOH**	**$Pb_2O(OH)_2$**	white		With $PbAc_2$ no ppt., Pb_2OAc_2 being sol.
2	**NaOH**	**$Pb(OH)_2$**	"	**NaOH** (ex.), **Acids**	
3	**Am_2CO_3**	**$Pb_3(CO_3)_2(OH)_2$**	"	**NaOH** (ex.), **HNO_3**	
4	**Na_2CO_3**	**$Pb_3(CO_3)_2(OH)_2$**	"	**NaOH** (ex.), **HNO_3**	
5	**HCl**	**$PbCl_2$**	"	**H_2O** (boiling) **HCl** (sl.)	Re-ppts., cryst., on cooling.
6	**H_2S**	**PbS**	black	dil. **HNO_3**	HNO_3 (str.) forms $PbSO_4$. $H_2S + HNO_3$ ppts. S.
7	**Am_2S**	**PbS**	"	dil. **HNO_3**	HNO_3 (str.) forms $PbSO_4$. $Am_2S + HNO_3$ ppts. S.
8	**KI**	**PbI_2**	yellow	**H_2O** boiling	Re-ppts., cryst., on cooling.
9	**H_2SO_4**	**$PbSO_4$**	white	**AmAc**, **NaOH** (ex.),	Dis. by $Na_2S_2O_3$ (v. $BaSO_4$).
10	**$K_2Cr_2O_7$**	**$PbCrO_4$**	yellow	**NaOH** (ex.), **HNO_3**	HCl changes ppt. to $PbCl_2$.
11	**Am_2Ox**	**PbOx**	white	**NaOH** (ex.), **HNO_3**	
12	**HNa_2PO_4**	**$Pb_3(PO_4)_2$**	"	**NaOH** (ex.), **HNO_3**	
13	**KCy**	**$PbCy_2$**	"	**KCy** (ex),	Boiling re-ppts.
14	**K_4FeCy_6**	**Pb_2FeCy_6**	"		
15	**Zn**	**Pb**	metallic	dil. **HNO_3**	Also Al, Cd, Co, Mg.
16	**Na_2CO_3** B.B.C.C.	**Pb**	"	dil. **HNO_3**	Yel. PbO on C.C.

49. SILVER, Ag′.

ANALYTICAL REACTIONS.

Directions.	Ppt.	Color.	Sol.
1. **Ppt. AgCl** (v. Pb, Hg′) To $AgNO_3$ sol. add HCl, filter (test fil.), wash res., reject fil. and washings.	(1) **AgCl**	white	
2. **Dis. AgCl in NH_4OH** (v. Hg′, Pb). To (1) add NH_4OH on filter, and catch fil. in t.t.			(a) $(NH_3)_2$ $(AgCl)_2$
3. **Re-ppt. AgCl.** Cautiously acidify (a) with HNO_3	(2) **AgCl**	white	

Supplementary. — 1. Strong HCl dis. AgCl (and HgCl somewhat); hence avoid excess. 2. In testing for *trace* of Ag, evap. NH_3 before adding HNO_3. 3. Never add the latter in large excess. AgCl is not re-ppd. by HCl except in conc. sols. (a) Sep. Ag from Pb. Are most salts of Ag soluble (Table A)? Can these exist together in sol: (b) $AgNO_3$ and $HgCl_2$? (c) $AgNO_3$ and $CuCl_2$?

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvent.	Remarks.
1	**AmOH**	Ag_2O	brown	**AmOH** (ex.), **KCy**, $Na_2S_2O_3$, **HAc**, H_2SO_4	AgOH is not known.
2	**NaOH**	"	"	"	
3	Am_2CO_3	Ag_2CO_3	white	Am_2CO_3 (ex.), **AmOH**, HNO_3	
4	Na_2CO_3	"	"	"	
5	**HCl**	**AgCl**	"	**AmOH**, **KCy**, $Na_2S_2O_3$	HNO_3 re-ppts. Light reduces to Ag_2Cl, violet, black.
6	H_2S	Ag_2S	black	boiling HNO_3	
7	Am_2S	"	"	"	
8	**KBr**	**AgBr**	white	**AmOH** (sl.)	
9	**KI**	**AgI**	yellow-white	**KI** (ex.)	Insol. in AmOH.
10	$K_2Cr_2O_7$	Ag_2CrO_4	dark red	**AmOH**, HNO_3	HCl changes it to AgCl.
11	Am_2Ox	Ag_2Ox	white	**AmOH**	
12	HNa_2PO_4	Ag_3PO_4	yellow	**AmOH**, HNO_3	
13	**KCy**	**AgCy**	white	**KCy** (ex.)	
14	K_4FeCy_6	Ag_4FeCy_6	yellow-white		Boiling gives metallic Ag.
15	**Zn**	**Ag**	gray	dil. HNO_3	Also Bi, Cu, Fe, Hg, Pb.
16	Na_2CO_3, B.B.C.C.	**Ag**	"	HNO_3	Metallic globule.

50. MERCURY (ous), Hg'.

ANALYTICAL REACTIONS.

Directions.	Ppt.	Color.	Sol.
1. Ppt. HgCl (v. Ag, Pb). To $HgNO_3$ sol. add HCl, filter (test fil.), wash, reject fil. and washings.	(1) **HgCl**	white	
2. AmOH on HgCl (v. Ag, Pb). On (1) pour a little AmOH, wash and reject washings.	(2) **NH_2Hg_2Cl**	black	
3. Dis. NH_2Hg_2Cl in aqua regia. On (2) pour a few drops aqua regia, save fil.			(a) **$HgCl_2$**
4. Re-ppt. HgCl. Dilute (a), add $SnCl_2$ sol.	(3) **HgCl** **Hg**	white gray	

Supplementary. — 1. Hg', not Hg'', must be used as $HgCl_2$ is sol. 3. The smallest quantity of *aqua regia* that will dis. the ppt. must be used. 4. $SnCl_2$ sol. must be newly prepared, and with plenty of HCl, or it will oxidize to $SnCl_4$ and Sn_2OCl_2. (a) Sep. Hg' from Ag. (b) Hg' from Pb. Explain action of $SnCl_2$ on $HgCl_2$. (c) Can $HgNO_3$ and $CuCl_2$ exist in sol. together? (d) $HgNO_3$ and $CdCl_2$?

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	**AmOH**	**$NH_2Hg_2NO_3$**	black	**HNO_3, aq.reg.**	Hg_2O (instable) = Hg + HgO. HgOH not known.
2	**NaOH**	**Hg_2O**	brown-black	" "	
3	**Am_2CO_3**	**$NH_2Hg_2NO_3$**	black	**Acids.**	
4	**Na_2CO_3**	**Hg_2CO_3**	gray		Forms black Hg_2O on boiling.
5	**HCl**	**HgCl**	white	**HNO_3**	With AmOH = NH_2Hg_2Cl (black).
6	**H_2S**	**Hg_2S**	black	**aq. reg.**	Hg_2S(instable) = HgS + Hg
7	**Am_2S**	"	"	"	
8	**KI**	**HgI**	yellow-green	**KI** (ex).	Ex. forms HgI_2(sol.) + Hg. Acids decompose it.
9	**H_2SO_4**	**Hg_2SO_4**	white	**HNO_3**	In strong sol. only.
10	**$K_2Cr_2O_7$**	**Hg_2CrO_4**	yellow	**HNO_3**	
11	**Am_2Ox**	**Hg_2Ox**	white	"	
12	**HNa_2PO_4**	**Hg_2PO_4**	"	"	
13	**KCy**	**$HgCy_2$ + Hg**	gray	**H_2O**	Hg ppd.
14	**K_4FeCy_6**	**Hg_4FeCy_6**	white	**HNO_3**	
15	**Zn.**	**Hg**	gray	**HNO_3, aq.reg.**	Also Al, Bi, Cd, Cu, Mg. Sn amalgamates with M.
16	**Na_2CO_3, B.B.C.C.**	**Hg**	"	" "	Heat in tube with Na_2CO_3 + C, and Hg sublimes.

51. GROUP I. — Pb″, Ag′, Hg′.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	SOL.
1. **Ppt. $PbCl_2$, AgCl, HgCl.** Sat. sol. with HCl, filter, wash, reject washings.	(1) **$PbCl_2$, AgCl, HgCl**	
2. **Sep. Pb from Ag, Hg.** On (1) pour boiling H_2O. Repeat till last fil. gives no ppt. with $K_2Cr_2O_7$ sol.	(2) **AgCl, HgCl**	(a) **$PbCl_2$**
3. **Final test for Pb.** To (a) add $K_2Cr_2O_7$ sol. (or $K_2Cr_2O_4$).	(3) **$PbCrO_4$**	
4. **Sep. Ag. from Hg.** On (2) pour warm AmOH two or three times.	(4) **NH_2Hg_2Cl**	(b) **$(NH_3)_2(AgCl)_2$**
5. **Final test for Ag.** Acidify (b) with HNO_3.	(5) **AgCl**	
6. **Dis. NH_2Hg_2Cl.** To (4) add very little aqua regia.		(c) **$HgCl_2$**
7. **Final test for Hg.** To (c) add $SnCl_2$ sol.	(6) **HgCl, Hg**	

Supplementary. — 1. Avoid large excess of HCl, as it may interfere with the ppts. of H_2S, Grp. II. AgCl is less sol. than HgCl, HgCl than $PbCl_2$. HCl may give a ppt. outside of Group I as follows: of S, if a sulfid or thiosulfate is present; of $BaCl_2$ (sat. sol.) which is sol. in H_2O but insol. in strong HCl; of BiOCl, SbOCl, Sn_2OCl_2, from sols. acidified with other acids than HCl (these are sol. in excess of HCl); of As_2S_3, Sb_2S_3, SnS_2, AgCy, $NiCy_2$, H_4SiO_4, H_3BO_3, $Zn(OH)_2$, $Al(OH)_3$, from an alk. sol. In any of the above cases if an appreciable ppt. falls, insol. in H_2O or HCl, dis. and analyze separately. 2. $PbCl_2$ should all be removed by successive additions of hot H_2O (test the last fil. with a drop of $AgNO_3$ sol.), as NH_4OH changes it to white insol. Pb_2OCl_2, some of which appears in the fil. HNO_3 however dis. it. It may be better to boil the liquid which holds the first ppt. before filtering, then filter and test the fil. for Pb. 3. What other reagent might be used here? 4. Why is not $AgNO_3$ formed? I have a white powder which is $PbCl_2$, HgCl, or AgCl. What reagent will determine which it is?

52. MERCURY (ic), Hg''.

ANALYTICAL REACTIONS.

Directions.	Ppt.	Color.	Sol.
1. **Ppt. HgS** (v. Pb, Bi, Cd, Cu, As, Sn, Sb). Sat. sol. with H_2S gas, filter (test fil.), wash.	(1) **HgS**	black	
2. **HgS insol. in Am_2S_x** (v. As, Sn, Sb). To (1) in e.d. add Am_2S_x, stir, boil, decant, wash.	(2) **HgS**	"	
3. **HgS insol. in dil. HNO_3** (v. Pb, Bi, Cd, Cu). Cover (2) with dil. HNO_3, boil a minute, decant.	(3) **HgS**	"	
4. **Dis. HgS in aqua regia** (v. Hg'). To (3) add little aqua regia, stir, boil if necessary.	(8)		(a) **$HgCl_2$**
5. **Ppt. HgCl and Hg with $SnCl_2$** (v. Hg', Sn''). Boil (a) to expel all Cl, add $SnCl_2$ sol.	(4) **HgCl Hg**	white gray	

Supplementary. — 1. Hg'' salts often give with H_2S a ppt. first white, then yellow, red, brown, black. Double salts., *e.g.* $HgCl_2(HgS)_2$ are first formed. 3. Strong HNO_3 may convert HgS into $Hg(NO_3)_2(HgS)_2$, white, insoluble; hence test a white res. for Hg. 4. In dis. HgS use the smallest quantity of aqua regia (usually only a few drops). The sol. may afterwards be diluted. 5. $SnCl_2$ sol. is pearly, and must not be confounded with a Hg ppt. How detect $HgNO_3$ in a mixture with $Hg(NO_3)_2$? $Hg(NO_3)_2$ in presence of $HgNO_3$? Explain the action of $SnCl_2$ on $HgCl_2$. Is HgS sol. in strong HNO_3? Why is Hg'' not put into Group I?

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	**AmOH**	**NH_2HgCl**	white	**AmOH, HCl**	$Hg(NO_3)_2$ gives NHg_2NO_3.
2	**NaOH**	**HgO**	yel.-red	**HNO_3**	$Hg(OH)_2$ not known.
3	**Am_2CO_3**	**NH_2HgCl**	white	**AmOH, HCl**	
4	**Na_2CO_3**	**$(HgO)_2HgCO_3$** (var.)	red white		Forms HgO on boiling.
5	**$SnCl_2$**	**HgCl, Hg**	gray	**HNO_3, aq. reg.**	
6	**H_2S**	**HgS**	black	**aqua regia**	May be first white, then red, brown, black.
7	**Am_2S**	"	"	"	
8	**KBr**	**$HgBr_2$**	white	**hot H_2O**	
9	**KI**	**HgI_2**	red	**$HgCl_2$ KI (ex.), HCl**	
10	**$K_2Cr_2O_7$**	**$HgCrO_4$**	orange	**HNO_3**	
11	**Am_2Ox**	**HgOx**	white	**dil. HNO_3**	With $HgCl_2$ no ppt. forms.
12	**HNa_2PO_4**	**$Hg_3(PO_4)_2$**	"	**acids, Am. salts**	
13	**KCy**	**$HgCy_2$**	"	**KCy (ex.)**	
14	**K_4FeCy_6**	**Hg_2FeCy_6**	"		On stand. becomes Pruss. blue.
15	**Zn**	**Hg**	gray		Forms amalgam with the metal (v. Exp. 50).
16	**Na_2CO_3** B.B.C.C.	**Hg**	"		

53. LEAD, Pb″.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. PbS** (v. Hg″, Bi, Cd, Cu, As, Sn, Sb). Sat. sol. with H_2S, filter (test fil.), wash.	(1) **PbS**	brown-black	
2. **PbS insol. in Am_2S_x** (v. As, Sn, Sb). To (1) in e.d. add Am_2S_x, warm, decant, wash.	(2) **PbS**	"	
3. **Dis. PbS in HNO_3** (v. Hg″). Boil (2) with dil. HNO_3, evap., add H_2O.			(a) **$Pb(NO_3)_2$**
4. **Ppt. $PbSO_4$** (v. Bi, Cd, Cu). To (a) add dil. $H_2SO_4 + C_2H_5OH$, and shake it.	(3) **$PbSO_4$**	white	

Supplementary. — 1. Pb sometimes gives colored ppts. at first, like Hg″, if much HCl is present. If there is much mineral acid, it must be diluted or partly neutralized before H_2S will give a ppt. 3. Strong HNO_3 oxidizes PbS to $PbSO_4$, and dil. acid does so somewhat. Some $PbSO_4$ dis. in HNO_3, and some may be left with HgS. Test the latter with boiling AmAc sol., filter and add $HAc + K_2Cr_2O_7$ sol. Before adding H_2SO_4 all HNO_3 should be expelled by evaporation. (a) Sep. Hg″ from Pb. Test the relative solubility in water of (b) $PbCl_2$, (c) PbS, (d) $PbSO_4$. Can these salts exist together in a sol.: (e) $Pb(NO_3)_2 + MgSO_4$? (f) $Pb(NO_3)_2 + Na_2CrO_4$? (g) $Pb(NO_3)_2 + NaCl$? Explain. Exp. 48 gives General Reactions for Pb.

54. BISMUTH, Bi‴.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. Bi_2S_3.** (v. Hg, Pb, Cd, Cu, As, Sb, Sn). Sat. sol. with H_2S, filter, wash.	(1) **Bi_2S_3**	brown-black	
2. **Bi_2S_3 insol. in Am_2S_x** (v. As, Sn, Sb). To (1) add Am_2S_x.	(2) **Bi_2S_3**	"	
3. **Dis. Bi_2S_3 in HNO_3** (v. Hg.). To (2) add dil. HNO_3, boil a minute, filter.	(S)		(a) **$Bi(NO_3)_3$**
4. **Bi not ppt. by H_2SO_4** (v. Pb). To (a) add few drops H_2SO_4.			(b) **$Bi(NO_3)_3$**
5. **Final test for Bi** (v. Cd, Cu). To (b) add excess AmOH, shake, let stand.	(3) **$Bi(OH)_3$**	white	

Supplementary. — 1. Diluting an acid sol. of Bi may ppt. BiOCl, $BiONO_3$, etc., but H_2S changes these to Bi_2S_3. 5. NH_4OH must be added in excess, otherwise Bi will not ppt. here, but with Cd (v. Group II, *A*). (a) Pour a few drops of the $BiCl_3$ sol. into a t.t. of H_2O. $BiCl_3$ reacts with H_2O, and forms insoluble BiOCl (v. Sb). (b) Test $Bi(NO_3)_3$ sol. in the same way. $BiNO_3(OH)_2$ (variable) is ppd. Name the ppts. Try to dis. them with acids: (c) HCl, (d) HNO_3, (e) HAc. Most salts of Bi react with H_2O. This confirmatory test may be tried in analysis. Ppt. (f) a Bi salt and (g) a Pb salt (in 2 tubes) with $K_2Cr_2O_7$ sol., and test solubility of each in NaOH sol. What would this reaction distinguish? Now add $SnCl_2$ sol. to each. (h) Sep. Bi from Hg″; (i) Bi from Pb; (j) Bi from Ag.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	**$Bi(OH)_3$**	white	**Acids**	Boil. gives Bi_2O_3 (yel.-wh.).
2	**NaOH**	"	"	"	No. ppt. with H_2Tr or H_3Cl.
3	**Am_2CO_3**	**$Bi_2O_2CO_3$**	"	"	
4	**Na_2CO_3**	"	"	"	
5	**$SnCl_2$**	**BiO**	black		With excess NaOH (v. Cl).
6	**H_2S**	**Bi_2S_3**	"	**HNO_3** (dil.)	Insol. in Am_2S_x.
7	**Am_2S**	"	"	"	" " "
8	**KI**	(BiOI) BiI_3	dark brown	**HCl**	
9	**H_2O**	**BiOCl**	white	**HCl**	Others give basic salt.
10 11	**$K_2Cr_2O_7$** or **K_2CrO_4**	**$Bi_2O(CrO_4)_2$**	yellow	**HNO_3**	Insol. in NaOH (v. Pb).
12	**Am_2Ox**	**Bi_2Ox_3**	white	**HNO_3**	
13	**HNa_2PO_4**	**$BiPO_4$**	"	**HCl**	Insol. in HNO_3 or HAc.
14	**KCy**	**$Bi(OH)_3$**	"	**Acids**	Insol. in excess KCy.
15	**K_4FeCy_6**	**Bi_4FeCy_{12}**	yellow-white	**HNO_3**	Reppts. on boiling.
16	**Zn**	**Bi**	spongy	"	Also Cu, Fe, Pb, Sn.
17	**Na_2CO_3** B.B.C.C.	**Bi**	dark	"	Incrustation yellow.

55. CADMIUM, Cd″.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. CdS** (v. Hg, Pb, Bi, Cu, As, Sb, Sn). Sat. sol. with H_2S; filter, wash.	(1) **CdS**	yellow	
2. **CdS insol. in Am_2S_x** (v. As, Sb, Sn). To (1) add Am_2S_x in e.d.; stir.	(2) **CdS**	yellow	
3. **Dis. CdS in HNO_3** (v. Hg). Boil (2) a minute with dil. HNO_3. Filter if necessary.	(S)		(a) **$Cd(NO_3)_2$**
4. **Cd not ppd. by H_2SO_4** (v. Pb). To (a) add a drop or two dil. H_2SO_4.			(b) **$Cd(NO_3)_2$**
5. **$Cd(OH)_2$ dis. by excess AmOH** (v. Bi). To (b) add excess. AmOH.			(c) **$(NH_3)_2CdO(AmNO_3)_2$**
6. **Cd not ppd. by KCy** (v. Cu). To (c) add little KCy sol.			(d) **$(KCy)_2CdCy_2$**
7. **Re-ppt. CdS** (v. Cu). Sat. (d) with H_2S.	(3) **CdS**	yellow	

Supplementary. — 1. If a large excess of acid is present the sol. must be diluted before H_2S will act. (a) Sep. Cd from Bi. (b) Sep. Cd from Hg″. (c) Sep. Cd from Pb. What sol. salts, if any, of Cd have the corresponding salts of Cu insol., or *vice versa* (v. Table A)? (e) Devise any method of separating Cd from Cu, dependent upon this, and try it.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	**$Cd(OH)_2$**	white	**AmOH** (ex)	
2	**NaOH**	"	"	**Acids**	Insol. in excess.
3	**Am_2CO_3**	**$CdCO_3$**	"		
4	**Na_2CO_3**	"	"	"	Free Am prevents. ppt.
5	**H_2S**	**CdS**	yellow	Hot, dil. **HNO_3**	
6	**Am_2S**	"	"	"	
7	**$K_2Cr_2O_7$**	**$CdCrO_4$**	"	**HNO_3**	In concentrated sols. only.
8	**Am_2Ox**	**CdOx**	white	**Acids** (sl.)	
9	**HNa_2PO_4**	**$Cd_3(PO_4)_2$**	"	**Acids**	
10	**KCy**	**$CdCy_2$**	"	**KCy** (ex.)	Re-ppd. by H_2S.
11	**K_4FeCy_6**	**Cd_2FeCy_6**	"	**HCl** **AmOH**	
12	**K_3FeCy_6**	**$Cd_3Fe_2Cy_{12}$**	yellow	**AmOH** **HCl**	
13	**Zn**	**Cd**	gray		Also Al, Mg.
14	**Na_2CO_3** B.B.C.C.	**CdO**	brown-yellow		Incrustation, no globule.

52. MERCURY (ic), Hg″.

ANALYTICAL REACTIONS.

Directions.	Ppt.	Color.	Sol.
1. **Ppt. HgS** (v. Pb, Bi, Cd, Cu, As, Sn, Sb). Sat. sol. with H_2S gas, filter (test fil.), wash.	(1) **HgS**	black	
2. **HgS insol. in Am_2S_x** (v. As, Sn, Sb). To (1) in e.d. add Am_2S_x, stir, boil, decant, wash.	(2) **HgS**	"	
3. **HgS insol. in dil. HNO_3** (v. Pb, Bi, Cd, Cu). Cover (2) with dil. HNO_3, boil a minute, decant.	(3) **HgS**	"	
4. **Dis. HgS in aqua regia** (v. Hg′). To (3) add little aqua regia, stir, boil if necessary.	(S)		(a) **$HgCl_2$**
5. **Ppt. HgCl and Hg with $SnCl_2$** (v. Hg′, Sn″). Boil (a) to expel all Cl, add $SnCl_2$ sol.	(4) **HgCl** **Hg**	white gray	

Supplementary. — 1. Hg″ salts often give with H_2S a ppt. first white, then yellow, red, brown, black. Double salts., *e.g.* $HgCl_2(HgS)_2$ are first formed. 3. Strong HNO_3 may convert HgS into $Hg(NO_3)_2(HgS)_2$, white, insoluble; hence test a white res. for Hg. 4. In dis. HgS use the smallest quantity of aqua regia (usually only a few drops). The sol. may afterwards be diluted. 5. $SnCl_2$ sol. is pearly, and must not be confounded with a Hg ppt. How detect $HgNO_3$ in a mixture with $Hg(NO_3)_2$? $Hg(NO_3)_2$ in presence of $HgNO_3$? Explain the action of $SnCl_2$ on $HgCl_2$. Is HgS sol. in strong HNO_3? Why is Hg″ not put into Group I?

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	**AmOH**	**NH_2HgCl**	white	**AmOH, HCl**	$Hg(NO_3)_2$ gives NHg_2NO_3.
2	**NaOH**	**HgO**	yel.-red	**HNO_3**	$Hg(OH)_2$ not known.
3	**Am_2CO_3**	**NH_2HgCl**	white	**AmOH, HCl**	
4	**Na_2CO_3**	**$(HgO)_2HgCO_3$** (var.)	red / white		Forms HgO on boiling.
5	**$SnCl_2$**	**HgCl, Hg**	gray	**HNO_3, aq. reg.**	
6	**H_2S**	**HgS**	black	**aqua regia**	May be first white, then red, brown, black.
7	**Am_2S**	"	"	"	
8	**KBr**	**$HgBr_2$**	white	**hot H_2O**	
9	**KI**	**HgI_2**	red	**$HgCl_2$** / **KI** (ex.), **HCl**	
10	**$K_2Cr_2O_7$**	**$HgCrO_4$**	orange	**HNO_3**	
11	**Am_2Ox**	**HgOx**	white	**dil. HNO_3**	With $HgCl_2$ no ppt. forms.
12	**HNa_2PO_4**	**$Hg_3(PO_4)_2$**	"	**acids, Am. salts**	
13	**KCy**	**$HgCy_2$**	"	**KCy** (ex.)	
14	**K_4FeCy_6**	**Hg_2FeCy_6**	"		On stand. becomes Pruss. blue.
15	**Zn**	**Hg**	gray		Forms amalgam with the metal (v. Exp. 50).
16	**Na_2CO_3** / **B.B.C.C.**	**Hg**	"		

52. MERCURY (ic), Hg''.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. HgS** (v. Pb, Bi, Cd, Cu, As, Sn, Sb). Sat. sol. with H_2S gas, filter (test fil.), wash.	(1) **HgS**	black	
2. **HgS insol. in Am_2S_x** (v. As, Sn, Sb). To (1) in e.d. add Am_2S_x, stir, boil, decant, wash.	(2) **HgS**	"	
3. **HgS insol. in dil. HNO_3** (v. Pb, Bi, Cd, Cu). Cover (2) with dil. HNO_3, boil a minute, decant.	(3) **HgS**	"	
4. **Dis. HgS in aqua regia** (v. Hg'). To (3) add little aqua regia, stir, boil if necessary.	(S)		(a) **$HgCl_2$**
5. **Ppt. HgCl and Hg with $SnCl_2$** (v. Hg', Sn''). Boil (a) to expel all Cl, add $SnCl_2$ sol.	(4) **HgCl** **Hg**	white gray	

Supplementary. — 1. Hg'' salts often give with H_2S a ppt. first white, then yellow, red, brown, black. Double salts., *e.g.* $HgCl_2(HgS)_2$ are first formed. 3. Strong HNO_3 may convert HgS into $Hg(NO_3)_2(HgS)_2$, white, insoluble; hence test a white res. for Hg. 4. In dis. HgS use the smallest quantity of aqua regia (usually only a few drops). The sol. may afterwards be diluted. 5. $SnCl_2$ sol. is pearly, and must not be confounded with a Hg ppt. How detect $HgNO_3$ in a mixture with $Hg(NO_3)_2$? $Hg(NO_3)_2$ in presence of $HgNO_3$? Explain the action of $SnCl_2$ on $HgCl_2$. Is HgS sol. in strong HNO_3? Why is Hg'' not put into Group I?

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	**NH_2HgCl**	white	**AmOH, HCl**	$Hg(NO_3)_2$ gives NHg_2NO_3.
2	**NaOH**	**HgO**	yel.-red	**HNO_3**	$Hg(OH)_2$ not known.
3	**Am_2CO_3**	**NH_2HgCl**	white	**AmOH, HCl**	
4	**Na_2CO_3**	**$(HgO)_2HgCO_3$** (var.)	red white		Forms HgO on boiling.
5	**$SnCl_2$**	**HgCl, Hg**	gray	**HNO_3, aq. reg.**	
6	**H_2S**	**HgS**	black	**aqua regia**	May be first white, then red, brown, black.
7	**Am_2S**	"	"	"	
8	**KBr**	**$HgBr_2$**	white	**hot H_2O**	
9	**KI**	**HgI_2**	red	**$HgCl_2$** **KI (ex.), HCl**	
10	**$K_2Cr_2O_7$**	**$HgCrO_4$**	orange	**HNO_3**	
11	**Am_2Ox**	**HgOx**	white	**dil. HNO_3**	With $HgCl_2$ no ppt. forms.
12	**HNa_2PO_4**	**$Hg_3(PO_4)_2$**	"	**acids, Am. salts**	
13	**KCy**	**$HgCy_2$**	"	**KCy (ex.)**	
14	**K_4FeCy_6**	**Hg_2FeCy_6**	"		On stand. becomes Pruss. blue.
15	**Zn**	**Hg**	gray		Forms amalgam with the metal (v. Exp. 50).
16	**Na_2CO_3** **B.B.C.C.**	**Hg**	"		

58. ARSENIC, As‴.

ANALYTICAL REACTIONS.

Directions.	Ppt.	Color.	Sol.
1. **Ppt. As_2S_3** (v. Group II, *A*; Sn, Sb). Sat. sol. with H_2S, filter (test fil.), wash.	(1) **As_2S_3**	yellow	
2. **As_2S_3 insol. in HCl** (v. Sn, Sb). Cover (1) with HCl, boil a minute, filter, wash.	(2) **As_2S_3**	yellow	
3. **Dis. As_2S_3 in Am_2S_x.** To (2) add Am_2S_x, boil, filter if necessary.	(S)		(a) **Am_3AsS_3**
4. **Re-ppt. As_2S_3.** Cautiously acidify (a) with HCl.	(3) **As_2S_3**, (S)	yellow	

Supplementary. — The original sol. should be acidified with H Cl. (a) Try a neutral sol. with H_2S, also (b) an alkaline. H_2S acts *very slowly* on As^v, but more rapidly when hot than when cold, ppg. As_2S_3+S. Test (c) an acid, (d) a neutral, (e) an alkaline sol. of As salts with a sol. of a Cu salt. What 2 yellow ppts. formed by H_2S? (f) Test the solubility of each in Am_2S_x.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	**$AgNO_3$**	**Ag_3AsO_3**	yellow	**AmOH**, **dil. HCl**, **AmCl**	As^v (neutral) + $AgNO_3$ = Ag_3AsO_4 (red-brown).
2	**H_2S**	**As_2S_3**	lemon-yellow	**Am_2CO_3**, **Am_2S_x**, **NaOH**	Re-ppd. by HCl.
3	**Am_2S**	"	"	"	
4	**$CuSO_4$**	**$HCuAsO_3$**	green	**AmOH**, **dil. acids**	Scheele's green (Paris green).
5	**$KMnO_4$**				Decolorized.
6	**$CuAc_2$**	**$CuO(As_2O_3)_3$**, **$CuAc_2$**	"	**AmOH** and acids	Schweinfurt green. Paris green.
7	**Cu**	**As**	gray	aq. reg.	Heated with HCl (Reinsch's test).
8	**H** (nascent)	**AsH_3** (gas)			Burns with pale-white flame. Deposits As on porcelain. AsH_3 very poisonous (Marsh's test).
9	**Na_2CO_3+KCy**	**As**	gray	HCl	Sublimed if heated dry.
10	**C** (heated)	"	"	"	Garlic odor.
11	**AmCl+AmOH +$MgSO_4$**	**$AmMgAsO_4$**	white	"	For As^v only.
12	**Am_2MoO_4**	**$AmAs(MoO_4)_2$**	yellow	"	Composition uncertain (v. M_3PO_4).

59. TIN, Sn″.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. SnS** (v. Group II, *A*; As, Sb). Sat. sol. with H_2S, filter, wash.	(1) **SnS**	chocolate	
2. **Dis. SnS in HCl** (v. As). To (1) add HCl, boil, filter if necessary.	(**S**)		(a) **$SnCl_2$**
3. **Hg test for Sn.** To (a) add $HgCl_2$ sol.	(2) **HgCl, Hg**	white gray	

Supplementary. — $SnCl_2$ sol. should be freshly prepared, and both Sn and HCl left in the sol. Otherwise $SnCl_4$ and Sn_2OCl_2 are formed. Sn^{iv} with H_2S gives yellow SnS_2, sol. in Am_2S_x, or Am_2CO_3. (a) Try to dis. Sn in HNO_3 (strong and dil.). Try to dis. part of the res. in (b) HCl, part in (c) aqua regia. What metals have no nitrates? Which of the following can exist in sol. together? (d) $SnCl_2$ and HgCl; (e) $SnCl_4$ and HgCl; (f) $SnCl_4$ and $HgCl_2$. (g) Change $SnCl_2$ to $SnCl_4$; (h) $SnCl_4$ to $SnCl_2$. Give proof of the change in both cases. (i) Sep. Sn from As. (j) Can $SnCl_2$ and $AgNO_3$ exist in sol. together? (k) $SnCl_2$ and $PbCl_2$?

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	**$Sn(OH)_2$**	white	**NaOH**	Ppt. from Sn^{iv} is $SnO(OH)_2$, white.
2	**NaOH**	"	"	"	Boiling gives SnO. Boiling NaOH + H_2Tr gives Sn (black).
3	**Am_2CO_3**	"	"	"	Insol. in excess (v. Sb‴).
4	**Na_2CO_3**	"	"	"	" " "
5	**$HgCl_2$**	**HgCl + Hg**	white, gray		If dil. HgCl, if strong Hg.
6	**H_2S**	**SnS**	brown	**NaOH**, **Am_2S_x**, **HCl**	Normal Am_2S does not dis. SnS. If Sn^{iv} is present, ppt. is SnS_2 (yellow).
7	**KI**	**SnI_2**	yellow	**HCl, NaOH**	In neutral, concentr. sol.
8	**Am_2Ox**	**SnOx**	white	**HCl**	
9	**HNa_2PO_4**	**$Sn_3(PO_4)_2$**	"	**Acids**	
10	**KCy**	**$Sn(OH)_2$**	"	**HCl, NaOH**	HCy (very poisonous) is evolved.
11	**K_4FeCy_6**	**Sn_2FeCy_6**	"		
12	**HNO_3, Cl**				Oxidizes Sn″ to Sn^{iv}.
13	**O or H_2O**	**Sn_2OCl_2**	white	**HCl**	$SnCl_4$ also formed and dissolved. Sn and HCl added retard action.
14	**Zn**	**Sn**	gray, spongy	"	Also Al, Cd, Mg.
15	**Na_2CO_3** B.B.C.C.	"			Use excess Na_2CO_3 and little KCy.

60. ANTIMONY, Sb'''.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. Sb_2S_3** (v. Group II, *A*; As, Sn). Sat. sol. with H_2S, filter, wash.	(1) **Sb_2S_3**	orange	
2. **Dis. Sb_2S_3 in HCl** (v. As). To (1) add HCl, boil, filter if necessary.			(a) **$SbCl_3$**
3. **Inaction of $HgCl_2$ on $SbCl_3$** (v. Sn). Divide (a), to half add $HgCl_2$ sol.			(b) **$SbCl_3$**
4. **Re-ppt. Sb_2S_3.** Dilute other half (a), add H_2S.	**Sb_2S_3**	"	

Supplementary. — Sb_2S_3 will not ppt. from too acid or too alkaline sols., and only imperfectly from neutral. $SbCl_3$ is insol. in H_2O, reacting with it to form insol. SbOCl (which H_2S changes to Sb_2S_3). It should be acidulated with HCl. Try to dis. Sb in HNO_3. If not sol., test the res. (a) with HCl, (b) with aqua regia. (c) Add $HgCl_2$ sol. to $SbCl_3$ sol. Explain. (d) Apply the "spot test," *i.e.* Marsh's test (Exp. 44), to Sb. (e) Sep. Sb from Sn; (f) Sb from As. Would you be likely to find $SbCl_3$ in sol. with the first group metals? With a purely aqueous sol. of Group II, *A*?

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	**Sb_2O_3**	white	**Hot NaOH** (ex.), **HCl**	Sol. should be slightly acid.
2	**NaOH**	"	"	" "	" " "
3	**Am_2CO_3**	"	"	" "	" " "
4	**Na_2CO_3**	"	"	**HCl**, **hot Am_2CO_3**	" " "
5	**H_2O**	**SbOCl** (var.)	"	**Acids**	Part only ppts.
6	**H_2S**	**Sb_2S_3**	orange	**Am_2Sx**, **NaOH**, **hot HCl**	
7	**Am_2S**	"	"	"	If too acid or too alkaline, no ppt.
8	**KI**	**no ppt.**	yellow		Sb^v ppts. I.
9	**Am_2Ox**	**$Sb(NH_3)_3Ox_3$**	white		Ppts. slowly.
10	**KCy**	**uncertain**	"		
11	**K_4FeCy_6**	"	"		Insol. in acids.
12	**Zn**	**Sb**	black	**aqua regia**	Also Bi, Cu, Mg, Fe, Sn.
13	**Na_2CO_3** B.B.C.C.	"	"	**aqua regia**	Metallic, brittle, white incrust., green flame, white fumes (Sb_2O_3).

61. GROUP II, *B*.—As, Sn, Sb.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	SOL.
1. **Ppt. As_2S_3, SnS, Sb_2S_3** (v. Group II, *A*). Sat. sol. with H_2S, filter, wash.	(1) **As_2S_3, SnS, Sb_2S_3**	
2. **Sep. Sn and Sb from As.** Warm (1) in e.d. some time with HCl, decant on filter.	(2) **As_2S_3** (S)	(a) **$SnCl_2$, $SbCl_3$**
3. **Dis. As_2S_3.** To (2) add Am_2S_x, boil, filter if necessary.	(S)	(b) **Am_3AsS_3**
4. **Final test for As.** Cautiously acidify (b) with HCl.	(3) **As_2S_3** (S)	
5. **Final test for Sn.** Divide (a), to half add $HgCl_2$ sol.	(4) **HgCl, Hg.**	
6. **Final test for Sb.** Dilute other half (a), add H_2S.	(5) **Sb_2S_3**	

Supplementary. — 1. If there is very much HCl in the sol., Sb and Sn may not ppt. To determine this, dilute a little of the sol., and test with H_2S. 2. The sol. should be warmed with HCl *in excess* some time, not boiled, or As will go into solution. As_2S_3 may be dis. by a sat. sol. of Am_2CO_3 (made without AmOH), warmed, and sep. from Sb and Sn; then ppt. As_2S_3 with HCl (cautiously). 3. If no appreciable res. is left in 2 except S, omit 3. 6. If Sb is present, an orange ppt. will appear first, followed later (if Sn is also there) by chocolate SnS. Metals of Group II, *B*, are sep. from those of Group II, *A*, by dis. the sulfids of the former in Am_2S_x (not Am_2S). A white ppt., on adding HCl is liable to be S. (a) Sep. $SbCl_3$ from $CuCl_2$; (b) $SnCl_2$ from $BiCl_3$.

62. IRON (ic), Fe‴.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. $Fe(OH)_3$** (v. Cr, Al). To sol. add AmCl sol., AmOH, warm, filter, wash res.	(1) **$Fe(OH)_3$**	red / brown	
2. **Dis. $Fe(OH)_3$ in HCl** (v. Cr, Al). To (1) in e.d. add dil. HCl, stir.			(a) **$FeCl_3$**
3. **Re-ppt. $Fe(OH)_3$ with NaOH** (v. Cr, Al). To (a) add excess NaOH sol.	(2) **$Fe(OH)_3$**	red / brown	

Supplementary. — To reduce Fe‴ to Fe″ v. Exp. 47. To oxidize Fe″ to Fe‴ v. Exp. 47. Test $FeCl_3$ sol. (Fe‴) in t.t. with a few drops of (a) K_4FeCy_6, (b) K_3FeCy_6, (c) KSCy, (d) NaOH. (e) Test also $FeSO_4$ sol. (Fe″) with the same reagents, and tabulate results. Then state what will distinguish Fe″ from Fe‴. (f) Dis. a little Fe in dil. H_2SO_4, nearly evap., add H_2O, and then determine whether $FeSO_4$ or $Fe_2(SO_4)_3$ is found. (g) Dis. a little Fe in aqua regia, evap., dil., test as before. (h) Test also the product after dis. in HCl. (i) Sep. Fe‴ from Cu″; (j) Fe″ from Bi‴. (It is better to oxidize Fe″ to Fe‴ before separation.) (k) Can a sol. contain both $FeCl_3$ and $HgNO_3$? (l) $FeSO_4$ and $Cu(NO_3)_2$? (m) Make $FePO_4$. Will H_2S ppt. FeS? Fe_2S_3 is not formed in aqueous analysis.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	**$Fe(OH)_3$**	red / brown	**Acids**	Variable to $Fe_2O_2(OH)_2$.
2	**NaOH**	"	"	"	" " "
3	**Am_2CO_3**	"	"	"	" " " Traces $Fe_2(CO_3)_3$.
4	**Na_2CO_3**	"	"	"	Variable to $Fe_2O_2(OH)_2$.
5	**$HNaPO_4$**	**$FePO_4$**	white	**M. acids**	Fe‴ (except $FeAc_3$) ppt. by this and NaAc (v. M_3PO_4).
6	**Am_2Ox**	**Fe_2Ox_3**	"	**Acids**	
7	**KCy**	**$Fe(OH)_3$**	red / brown	"	HCy formed (very poisonous).
8	**K_4FeCy_6**	**$Fe_4Fe_3Cy_{18}$**	blue	**H_2SO_4**	Prussian blue. NaOH + ppt. = $Fe(OH)_3$.
9	**K_3FeCy_6**				No action; distinguishes Fe‴ from Fe″.
10	**KSCy**	**$FeCy_3S_3$**	blood-red		Color only, no ppt. Disting. Fe‴ from Fe″.
11	**H_2S**	**S**	white		Reduces Fe‴ to Fe″.
12	**Am_2S**	**FeS + S**	black	**Acids**	First reduces Fe‴ to Fe″.
13	**Tannic acid and galls.**	**Fe‴ tannate**	"		Basis of writing ink.
14	**NaAc**	**$Fe_2Ac_5(OH)_3$**	red		
15	**SO_2, H_2S / Zn, $SnCl_2$**				Reduce Fe‴ to Fe″.

63. IRON, Fe″.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$AmOH$	$Fe(OH)_2$	green, black	$AmCl$ (sl.), Acids	White, if pure. Oxidizes to $Fe(OH)_3$, red-brown.
2	$NaOH$	"	"	"	" " "
3	Am_2CO_3	$FeCO_3$	white	"	Oxidizes to $Fe(OH)_3$.
4	Na_2CO_3	"	"	"	" " "
5	H_2S	FeS	black	"	With $FeAc_2$ only a slight ppt.
6	Am_2S	"	"	"	Oxidizes to $FeO(SO_4)_2$.
7	Am_2Ox	$FeOx$	yellow-white	M. acids	
8	HNa_2PO_4	$HFePO_4$	blue-white	Acids	$Fe_3(PO_4)_2$ also formed.
9	KCy	$FeCy_2$	yellow-red	KCy (ex.)	
10	K_4FeCy_6	$K_2Fe_2Cy_6$	blue-white		Oxidizes in air to Prussian blue.
11	K_3FeCy_6	$Fe_3Fe_2Cy_{12}$	blue		Distinguishes Fe″ from Fe‴.
12	$KSCy$	no effect			" " "
13	HNO_3, $KClO_3$				
14	Cl, &c.	Fe‴ (no ppt.)	red		Oxidizes Fe″ to Fe‴.
15	Tannic acid + tincture galls	Fe″ tannate	white to black		Oxidizes to Fe‴ tannate (black ink).

Supplementary. — In the separation of Fe″ it is first oxidized to Fe‴, and then tested as in Group III. To ascertain whether you have a ferrous (Fe″) or a ferric (Fe‴) salt, test the original sol. by the table below. (a) Prepare some Prussian blue; (b) Everitt's salt; (c) Turnbull's blue; (d) black ink.

TESTS FOR FERROUS AND FERRIC IRON.

Re-agt.	Fe″.	Fe‴.
K_4FeCy_6	$K_2FeFeCy_6$ (bluish white). Potassium ferrous-ferro-cyanid or Everitt's salt.	$Fe_4Fe_3Cy_{18}$ (deep-blue). Ferric-ferro-cyanid or Prussian blue.
K_3FeCy_6	$Fe_3Fe_2Cy_{12}$ (dark-blue). Ferrous-ferri-cyanid or Turnbull's blue.	$FeFeCy_6$ (green or brown color only). Ferric-ferri-cyanid.
$KSCy$	No effect.	$Fe(SCy)_3$ (blood-red color only). Ferric-sulpho-cyanid.
KOH	$Fe(OH)_2$ (white to dirty green). Ferrous hydroxid.	$Fe(OH)_3$ (red-brown). Ferric hydroxid.

64. ALUMINUM, Al'''.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. $Al(OH)_3$** (v. Fe, Cr). To sol. add AmCl sol., AmOH, boil, filter.	(1) **$Al(OH)_3$**	white	
2. **Dis. $Al(OH)_3$ in HCl** (v. Fe, Cr). To (1) add dil. HCl, stir.			(a) **$AlCl_3$**
3. **Dis. $Al(OH)_3$ in NaOH** (v. Fe, Cr, Zn). To (a) add excess. strong NaOH sol., shake.			(b) **$Na_2Al_2O_4$**
4. **$Al(OH)_3$ insol. in AmOH.** Acidify (b) with HCl, add AmOH.	(2) **$Al(OH)_3$**	"	

Supplementary. — 3. NaOH sol. must be in excess, and strong enough to dis. the $Al(OH)_3$ first formed. (a) Sep. Fe from Al. (b) Test action of limited NaOH sol. on Al. (c) Is Al_2S_3 formed in aqueous analysis [try H_2S and Am_2S; a ppt. is $Al(OH)_3$]? (d) Test Al''' and AmCl sol. with NaOH sol. (e) Now ppt. $Al(OH)_3$ with AmCl and NaOH, and try to dis. it in excess. (f) Sep. Cu and Al; (g) Bi and Al. (h) Obtain $AlCl_3$ from several cpds. of Al, in two or three different ways. (i) Obtain alum crystals by mixing sols. of sulfates of Al and K (Exp. 5). Try to dis. Al in (j) HCl, (k) HNO_3.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	**$Al(OH)_3$**	gray, white	**NaOH, acids**	NaOH (ex.) forms $Na_2Al_2O_4$, not re-ppd. by boiling (v. Zn).
2	**NaOH**	"	"	"	NaOH (ex.) forms $Na_2Al_2O_4$; AmCl re-ppts. it.
3	**Am_2CO_3**	"	"	**Na_2CO_3**	Liberates CO_2.
4	**Na_2CO_3**	"	"	**Na_2CO_3** (ex.)	" "
5	**Am_2S**	"	white	**Acids**	H_2S evolved.
6	**K_4FeCy_6**	**uncertain**	"	"	
7	**HNa_2PO_4**	**$AlPO_4$**	"	**NaOH, HCl**	
8	**$Na_2S_2O_3$**	**$Al(OH)_3 + S$**	"	**Acids**	SO_2 liberated.
9	**$Na_2B_4O_7$** (bead) B.B.C.C. + $Co(NO_3)_2$		blue		First heat: infus. Add a drop of $Co(NO_3)_2$ sol., heat: blue.

65. CHROMIUM (ic), Cr'''.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. $Cr(OH)_3$** (v. Fe, Al). To sol. add AmCl sol., AmOH; warm.	(1) **$Cr(OH)_3$**	green	
2. **Dis. $Cr(OH)_3$ in HCl** (v, Fe, Al). To (1) add dil. HCl, stir, filter if necessary.			(a) **$CrCl_3$**
3. **Oxidize Cr''' to Cr^{vi}.** Evap. (a) almost to dryness, then add 1 or 2 crystals $KClO_3$, and evap. to dryness; add H_2O, filter if necessary.			(b) **$K_2Cr_2O_7$**
4. **Action of NaOH on $K_2Cr_2O_7$.** To (b) add NaOH sol.			(c) **K_2CrO_4**
5. **Ppt. $PbCrO_4$.** Acidify (c) with HAc, add $Pb(NO_3)_2$ sol.	(2) **$PbCrO_4$**	yellow	

Supplementary. — 1. $Cr(OH)_3$ may dis. in cold water, but re-ppts. on boiling. 3. Cr''' must be oxidized to Cr^{vi}, or it will ppt. with NaOH sol. (a) Sep. Cr''' from Al'''. (b) To $K_2Cr_2O_7$ sol. add successive small portions of AmOH, shaking each time, till the color becomes yellow. Alkalies reduce red dichromates to yellow chromates; acids have the reverse effect. (c) Slowly acidify the chromate with HCl. (d) Will $Cr(OH)_3$ ppt. from $K_2Cr_2O_7$ sol.? (e) Reduce Cr^{vi} to Cr'''. (f) Sep. Cr''' and Fe'''; (g) Cr''' and Cd. (h) Make $Cr_2(SO_4)_3$ from $K_2Cr_2O_7$. (i) Make $Cr(NO_3)_3$.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	**$Cr(OH)_3$**	blue-green	**NaOH** (ex.) **Acids**	Re-ppt. by hot AmCl or by boiling.
2	**NaOH**	"	"	**NaOH** (ex.)	" " " "
3	**Am_2CO_3**	"	"	**Na_2CO_3** (ex.)	" " " "
4	**Na_2CO_3**	"	"	"	" " " " CO_2 formed. Slight ppt.
5	**H_2S**	**$Cr_2O_2CrO_4$**			Reduces Cr^{vi} to Cr''' in presence of HCl.
6	**Am_2S**	**$Cr(OH)_3$**	"	**Acids**	Forms slowly if dilute; H_2S liberated.
7	**HNa_2PO_4**	**$CrPO_4$**	green	**M. acids**	
8	**$H_2SO_4+H_2O_2$**	**undeterm.**	blue	**$(C_2H_5)_2O$**	Blue liquid rises to surface with Cr^{vi} only.
9	**KCy**	**$Cr(OH)_3$**	blue-green	**NaOH**	HCy liberated.
10	**$KClO_3$, Cl, &c.**				Cr''' oxidized to Cr^{vi} (yellow-red) with heat.
11	**$Na_2CO_3+KClO_3$**	**Na_2CrO_4 K_2CrO_4**	yellow	**H_2O**	Fused on Pt foil.
12	**Na_2CO_3** (bead)		green		V. Table G.

66. GROUP III.—Fe, Al, Cr.

SEPARATION.

DIRECTIONS.	PPT.	SOL.
1. **Ppt. $Fe(OH)_3$, $Al(OH)_3$, $Cr(OH)_3$.** To sol. add AmCl sol., AmOH, boil a minute, filter, wash.	(1) **$Fe(OH)_3$, $Al(OH)_3$, $Cr(OH)_3$**	
2. **Dis. $Fe(OH)_3$, $Al(OH)_3$, $Cr(OH)_3$ in HNO_3.** To (1) add dil. HNO_3.		(a) **$Fe(NO_3)_3$, $Al(NO_3)_3$, $Cr(NO_3)_3$**
3. **Oxidize $Cr(NO_3)_3$ to $K_2Cr_2O_7$.** Evap. (a) to dryness; when almost evap. add 1 or 2 small crystals $KClO_3$. When evap. dis. in H_2O; decant on filter and add to res. a *few drops* HNO_3; pour on same filter.		(b) **$Fe(NO_3)_3$, $Al(NO_3)_3$, $K_2Cr_2O_7$**
4. **Sep. Fe from Al and Cr.** To (b) add excess strong NaOH sol., shake, filter.	(2) **$Fe(OH)_3$**	(c) **$Na_2Al_2O_4$, K_2CrO_4**
5. **Sep. Al from Cr.** Acidify (c) with HCl, add AmOH, filter.	(3) **$Al(OH)_3$**	(d) **K_2CrO_4**
6. **Final test for Cr.** Partly evap. (d), dil., acidify with HAc, add $Pb(NO_3)_2$ sol.	(4) **$PbCrO_4$**	

Supplementary. — 1. AmCl prevents ppn. by AmOH of Mg, Mn, Zn, Co, Ni (Fe″ partially), aids ppn. of Al, and does not affect Fe‴ and Cr. Considerable AmCl is needed. AmOH at first ppts. Co, Ni, Zn, if present, all of which are sol. in excess. If only one of these Fe‴, Al, Cr, is present, the color determines which. $Cr(OH)_3$ dis. in cold H_2O, but is re-ppd. on boiling. Fe″ should be oxidized to Fe‴ by boiling with a *few drops* of HNO_3. H_2S must all be removed by boiling before adding AmOH, to prevent formation of Am_2S and ppn. of Co, Ni, Mn, Zn. Cr^{vi} is reduced by H_2S to Cr‴, and hence ppd. here. 3. Cr‴ must all be oxidized to Cr^{vi} or it will ppt. with Fe. 4. Fe should be tested in the original sol. for Fe″ or Fe‴ by K_4FeCy_6, K_3FeCy_6, KSCy. 5. NaOH dis. some SiO_2 from the containing bottles, and there may be a ppt. of H_4SiO_4, similar to $Al(OH)_3$, small in quantity and sinking sooner than $Al(OH)_3$. 6. A white ppt. of $PbCl_2$ will always appear if no Cr is present. The yellow color alone indicates Cr.

67. COBALT, Co″.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. CoS** (v. Ni, Mn, Zn). To sol. add AmCl sol., Am_2S, shake, warm, filter, wash res. with hot H_2O, adding to it a few drops Am_2S.	(1) **CoS**	black	
2. **CoS insol. in HCl** (v. Mn, Zn). To (1) add dil. HCl, stir, decant.	(2) **CoS**	"	
3. **Dis. CoS in aqua regia** (v. Ni). To (2) add a few drops of aqua reg., stir, filter, evap. acid, dil.	(S)		(a) $CoCl_2$
4. **Ppt.** $K_6Co_2O(NO_2)_{10}$. To (a) add HAc, KNO_2 sol.	(3) $K_6Co_2O(NO_2)_{10}$	yellow	

Supplementary. — 1. Am_2S (colorless) must be used, not Am_2S_x (yellow). 2. CoS is sol. in strong, hot HCl, and slightly in cold, as is NiS. 3. $K_6CoO(NO_2)_{10}$ is broken up by alkalies, or HCl, but not by $HC_2H_3O_2$. 1. CoS and NiS are liable to oxidize to the soluble sulfates if exposed long to the air, hence they must be filtered and washed at once. 4. Some ppt. may occur at once in 4, but to be sure all the Co is thrown down 24 hours is needed, and enough KNO_2 must be used. What sulfid in Group II was sol. in aqua regia only? Why is not CoS ppd. by H_2S with Group II? (a) To Co salts add H_2S, then AmOH. How is Am_2S made? (b) Make a borax bead by fusing with the blow-pipe a little $Na_2B_4O_7$ in the loop of a Pt. wire until it becomes clear. Dip this bead into a very weak sol. of $CoCl_2$ and fuse again. The blue color — if the sol. was not too strong — is very characteristic of Co. (c) Can $AgNO_3$ and $CoCl_2$ exist in sol. together? Sep. (d) Co and Cd; (e) Co and Cr.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	$Co(OH)_2$	red	**AmOH** (ex.), **AmCl, &c.**	Blue bas. ppt. first formed. On stand. forms $Co(OH)_2$. Oxidizes to $Co(OH)_3$.
2	**NaOH**	"	"	"	Insol. in NaOH (ex.).
3	Am_2CO_3	Co_2OCO_3	"	Am_2CO_3 (ex.)	Violet or blue by heating.
4	Na_2CO_3	"	"		Insol. in excess Na_2CO_3.
5	Am_2S	**CoS**	black	HNO_3, **aq. reg.**	Oxid. to $CoSO_4$, which is sol.
6	KNO_2	$K_6Co_2O(NO_2)_{10}$	yellow	**HCl**	Ppts. only in pres. of HAc.
7	**KCy**	$CoCy_2$	br.-wh.	**KCy** (ex.)	Dist. from Ni by hot HCl.
8	K_4FeCy_6	Co_2FeCy_6			Grayish green.
9	K_3FeCy_6	$Co_3Fe_2Cy_{12}$	brown-red		In absence of Mn″, or Ni, AmCl+AmOH+K_3FeCy_6 +Co″ = blood-red color.
10	HNa_2PO_4	$HCoPO_4$	red	**AmOH, acids**	
11	**Heat** (on paper)		blue		Dehydrated.
12	$Na_2B_4O_7$ (bead)		"		Blue bead in either flame.
13	**Cl**, PbO_2 (warm)		white		Oxidizes Co″ to Co‴.
14	**Zn**	**Co**	"	**HCl**	Also Cd, Mg.
15	Na_2CO_3 B.B.C.C.	"	"	"	Magnetic.

68. NICKEL, Ni″.

ANALYTICAL REACTIONS.

Directions.	Ppt.	Color.	Sol.
1. **Ppt. NiS** (v. Co, Mn, Zn). To sol. add AmCl, Am_2S, warm, filter rapidly, wash res. with hot H_2O and a few drops of Am_2S.	(1) **NiS**	black	
2. **NiS insol. in HCl** (v. Mn, Zn). To (1) add little dil. HCl, stir and decant.	(2) **NiS**	"	
3. **Dis. NiS in aqua regia** (v. Co). To (2) add at once a few drops of aqua regia, stir, filter, evap. acid, and dilute.	(3)		(a) $NiCl_2$
4. **Inaction of KNO_2 on NiCl and HAc.** To (a) add little HAc, KNO_2 sol.; leave 24 hours.			(b) $NiCl_2$
5. **Ppt. $Ni(OH)_2$.** To (b) add *excess strong* NaOH sol.; boil if necessary.	(3) $Ni(OH)_2$	apple-green	

Supplementary. — 1. Ni has not been wholly removed if the fil. is brown or has black particles in it. In that case acidify the fil. with HAc, boil, filter, and test for Ni. 3. In using aqua regia the smallest quantity (usually a few drops) that will suffice to dis. the substance must be employed. The sol. may afterwards be diluted. Note the color of Ni salts. Sep. (a) Ni and Co; (b) Ni and Fe; (c) Ni and Cu; (d) Ni, Cu, Al; (e) Ni, Bi, Sb. (f) Test Ni with borax bead (v. Co and Table G).

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	**AmOH**	$Ni(OH)_2$	pale green	**AmOH**, **AmCl**, &c.	Sol. NaOH re-ppts. slowly (violet-blue).
2	**NaOH**	"	"	"	Insol. in NaOH (ex.). Unchanged in air.
3	Am_2CO_3		green	Am_2CO_3 (ex.)	Sol. has blue-green color.
4	Na_2CO_3	"	"	Na_2CO_3 (ex.)	" " " "
5	H_2S	**NiS**	black	HNO_3, aq. reg.	From neutral or alk. sol., not acid.
6	Am_2S	"	"	"	Sol. (sl.) in Am_2S (ex.).
7	K_4FeCy_6	Ni_2FeCy_6	green-white	**AmOH**	
8	K_4FeCy_6	$Ni_2Fe_2Cy_{12}$	green-yel.		AmCl+AmOH+K_4FeCy_6+Ni″, boiled = red ppt.
9	HNa_2PO_4	$Ni_3(PO_4)_2$	green-white		
10	**KCy**	$NiCy_2$	yel.-green	**KCy** (ex.)	Dist. from Co by HCl.
11	M_2Ox	**NiOx**	green		Leave 24 hours.
12	$Na_2B_4O_7$ (bead)				(V. Table G.) Co″ obscures test.
13	Cl, Br, &c.				Oxidizes Ni″ to Ni‴.
14	H_2S, SO_2, &c.				Reduces Ni‴ to Ni″.
15	**Zn**	**Ni**	white		Also Cd, Sn.
16	Na_2CO_3 B.B.C.C.	"	"	HNO_3	Magnetic powder.

69. MANGANESE (ous), Mn''.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. MnS** (v. Co, Ni, Zn). To sol. add AmCl sol., Am_2S, warm, filter, wash in hot H_2O and Am_2S.	(1) **MnS**	salmon	
2. **Dis. MnS in HCl** (v. Co, Ni). To (1) add dil. HCl, stir, filter, boil to expel all H_2S, NH_3, &c. (test a little with $PbAc_2$).	(S)		(a) **$MnCl_2$**
3. **Ppt. $Mn(OH)_2$** (v. Zn). To (a) add excess strong NaOH sol., warm.	(2) **$Mn(OH)_2$** **$Mn_2O_2(OH)_2$**	white brown	

Supplementary. — 3. $Mn(OH)_2$ will not ppt. with NaOH if AmCl and AmOH are present. $Mn(OH)_2$ when pure is white, but it rapidly oxidizes to brown $Mn_2O_2(OH)_2$ (variable). This should further be tested by fusing it with a mixture of Na_2CO_3 and KNO_3, when bright green K_2MnO_4 and Na_2MnO_4 are formed. (a) See whether $Mn(OH)_2$ or MnS forms in preference to the other. Will these exist together in sol.: (b) $MnCl_2 + ZnSO_4$? (c) $MnSO_4 + Cu(NO_3)_2$? (d) $MnCl_2 + K_2CrO_4$? Sep. (e) Mn and Ni; (f) Mn and As; (g) Mn, Cu, Cr; (h) Pb, Sr, Mn.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**AmOH**	**$Mn(OH)_2$**	white	**AmCl, &c.**	Quickly oxidizes to brown $Mn_2O_2(OH)_2$; insoluble in AmCl.
2	**NaOH**	"	"	"	" " " "
3	**Am_2CO_3**	**$MnCO_3$**	"	"	" " " "
4	**Na_2CO_3**	"	"	"	" " " "
5	**H_2S**	**MnS**	pink	**HCl, HAc**	MnS ppts. from $MnAc_2$ (unless HAc is present). Not otherwise.
6	**Am_2S**	"	"	"	
7	**K_4FeCy_6**	**Mn_2FeCy_6**	white	**HCl**	
8	**K_3FeCy_6**	**$Mn_3Fe_2Cy_{12}$**	brown		Insol. in acids.
9	**HNa_2PO_4**	**$Mn_3(PO_4)_2$**	white	**Dil. acids**	From Am_2MnCl_4, etc., forms $AmMnPO_4$.
10	**KCy**	**$MnCy_2$**	"	**KCy** (ex.)	Darkens in air.
11	**M_2Ox**	**MnOx**	"	**Acids**	
12	**$Na_2B_4O_7$** (bead)		amethyst		In o. f. In r. f. colorless (v. Table G).
13	**Na_2CO_3+KNO_3** ignited on Pt foil	**K_2MnO_4** **Na_2MnO_4**	green		Na_2CO_3 bead (o. f.), same green color.
14	**Cl, Br, &c.**				Oxidizes to Mn'''.
15	**(H_2S), SO_2, &c.**				Reduces Mn''' to Mn'', ppts. S.
16	**Zn**				Reduces Mn''' to Mn'' when rightly acidulated.
17	**HNO_3+PbO_2** (boil)	**$HMnO_4$** (sol.)	red		Seen on settling of sediment.

70. ZINC, Zn″.

ANALYTICAL REACTIONS.

Directions.	Ppt.	Color.	Sol.
1. **Ppt. ZnS** (v. Co, Ni, Mn). To sol. add AmCl, Am_2S, warm, filter, wash with hot H_2O and a few drops of Am_2S.	(1) **ZnS**	white	
2. **Dis. ZnS in HCl** (v. Co, Ni). To (1) add dil. HCl, stir, filter.	(S)		(a) **$ZnCl_2$**
3. **$Zn(OH)_2$ sol. in excess NaOH** (v. Mn). To (a) add excess strong NaOH sol.			(b) **Na_2ZnO_2**
4. **Re-ppt. ZnS.** Acidify (b) with HAc, add Am_2S.	(2) **ZnS**	"	

Supplementary. — 1. ZnS is the only white sulfid, but it must not be confounded with a white ppt. of S. 3. $Zn(OH)_2$ is not sol. in dil. sol. of NaOH, especially if boiled. 4. ZnS is ppd. by H_2S if the sol. is acidified with HAc. Zn ppts. these metals from sols. of their salts: Pb, Ag, Hg, Bi, Cd, Cu, As, Sn, Sb, Co, Ni, Fe. State color of each sulfid in Groups II and IV (tabulate). What hydroxids are sol. in excess of NaOH? Sep. (a) Zn and Al; (b) Zn and Co; (c) Zn and Mn; (d) Pb, Fe, Zn.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	**AmOH**	$Zn(OH)_2$	gel. white	**AmOH** (ex.)	Forms Am_2ZnO_2. Boiling re-ppts.
2	**NaOH**	"	"	**NaOH** (ex.)	All Zn salts are sol. in NaOH, except ZnS.
3	Am_2CO_3	$Zn_5(OH)_6(CO_3)_2$	"	**AmOH**	
4	Na_2CO_3	"	"	"	
5	H_2S	**ZnS**	white		From neutral sol. only, and in part only.
6	Am_2S	"	"	**HCl**	Insol. in HAc (v. Mn″).
7	K_4FeCy_6	Zn_2FeCy_6	"		
8	K_4FeCy_6	$Zn_3Fe_2Cy_{12}$	yellow		
9	**KCy**	$ZnCy_2$	white	**KCy** (ex.)	
10	$Na_2B_4O_7$ (bead)				Hot, yellow; cold, white.
11	Na_2CO_3 B.B.C.C.	**ZnO, Zn**	yellow		As incrustation. On cooling becomes white. $Co(NO_3)_2$ makes green color on heating again.

71. GROUP IV.—Co, Ni, Mn, Zn.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	SOL.
1. **Ppt. CoS, NiS, MnS, ZnS.** To sol. add AmCl, Am_2S, shake, warm (not boil), filter rapidly. Pour on res. a few drops Am_2S, then wash with hot H_2O.	(1) **CoS, NiS, MnS, ZnS**	
2. **Sep. Co, Ni from Mn, Zn.** To (1) in e.d. add dil. HCl, stir well, filter and wash.	(2) **CoS, NiS**	(a) **$MnCl_2$, $ZnCl_2$**
3. **Dis. CoS and NiS.** To (2) add at once *very little aqua regia*. Evap. acid, dil., add HAc.	(3)	(b) **$CoCl_2$, $NiCl_2$**
4. **Sep. Co from Ni.** To (b) add excess strong KNO_2 sol., leave 24 hours, filter.	(3) **$K_6Co_2O(NO_2)_{10}$**	(c) **$NiCl_2$**
5. **Final test for Ni.** To (c) add *excess strong* NaOH sol., boil.	(4) **$Ni(OH)_2$**	
6. **To separate Mn from Zn.** Partly evap. (a) to concentrate the sol. and expel all NH_3, acidify with HAc, add excess strong NaOH sol., shake, let stand, filter.	(5) **$Mn(OH)_2$, $Mn_2O_3(OH)_2$**	(d) **Na_2ZnO_2**
7. **Final test for Zn.** Acidify (d) with HAc, add Am_2S.	(6) **ZnS**	

Supplementary. — 1. Am_2S (colorless), not Am_2S_x (yellow), must be used. CoS, NiS, etc., oxidize to sulfates in air. Hence filter rapidly with H_2O having a few drops of Am_2S. Group II metals, if not all removed, may ppt. here. 2. CoS, NiS are dis. by strong hot HCl, and slightly by cold. 6. ZnS, MnS will ppt. on adding NaOH if any H_2S is present. Mn will not ppt. with NaOH if AmCl and AmOH are present. $Zn(OH)_2$ is sol. in excess of strong cold NaOH sol. only. Mn should be tested for by fusing with Na_2CO_3 and KNO_3. (a) What members of Group III will Am_2S ppt.? (b) of Group II? (c) Group I?

72. BARIUM, Ba''.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Inaction of AmCl, AmOH on Ba salts** (v. Mg). To sol. add AmCl sol., AmOH.			(a) $BaCl_2$
2. **Ppt. $BaCO_3$** (v. Sr, Ca, Mg). To (a) add Am_2CO_3 sol., warm, filter, wash.	(1) $BaCO_3$	white	
3. **Dis. $BaCO_3$ in HAc** (v. Sr, Ca). To (1) add little HAc.			(b) $BaAc_2$
4. **Action of $K_2Cr_2O_7$ on $BaAc_2$** (v. Sr, Ca). To (b) add $K_2Cr_2O_7$ sol.	(2) $BaCrO_4$	yellow	

Supplementary. — Has AmOH any effect on sol. salts of Ba? (a) Sep. Ba and Mn; (b) Ba and Fe'''; (c) Ba and Bi; (d) Ba and Hg''; (e) Ba and Hg'. (f) What numbers of Group IV will Am_2CO_3 ppt.? (g) Group III? (h) Group II? (i) Group I? What Ba salt is insol. in HCl (v. Exp. 100)? For what test is $Ba(OH)_2$ used? $BaCO_3$ occurs free in nature. How can other Ba salts be made from it? (g) Make several. (h) Test the Ba flame by dipping a Pt. wire (or a splinter) into $BaCl_2$ and holding it in the edge of a Bunsen flame.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**NaOH**	$Ba(OH)_2$	white	**20 H_2O, Acids**	Must be very conc. sol.
2	Am_2CO_3	$BaCO_3$	"	**Acids**	Slightly sol. in AmCl, aided by AmOH and heat.
3	Na_2CO_3	"	"	"	" " " " "
4	HNa_2PO_4	$HBaPO_4$	"	"	Also AmCl (sl.). Must be neutral or alkaline.
5	M_2SO_4	$BaSO_4$	"	**350,000 H_2O**	One of the most insol. of salts.
6	K_2CrO_4	$BaCrO_4$	yellow	**HAc (sl.), HCl (warm)**	Re-ppd. by AmOH.
7	$K_2Cr_2O_7$	"	"	"	" " "
8	H_2SiF_6	$BaSiF_6$	white	**HCl, HNO_3**	C_2H_5OH aids ppn., also stirring, standing and boiling. Sr not ppd. by H_2SiF_6.
9	Am_2Ox	**BaOx**	white	**Acids**	From conc. sols.
10	**Flame test**		yellow-green		Vis. through indigo sol.; blue-green through green glass.

73. STRONTIUM, Sr''.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COL.	SOL.
1. **Inaction of AmCl and AmOH on Sr salts** (v. Mg). To sol. add AmCl sol., AmOH.			(a) $SrCl_2$
2. **Ppt. $SrCO_3$** (v. Mg, Ba, Ca). To sol. add Am_2CO_3 sol., warm, filter, wash.	(1) $SrCO_3$	white	
3. **Dis. $SrCO_3$ in HAc** (v. Ba, Ca). Pour on (1) a little HAc.			(b) $SrAc_2$
4. **Inaction of $K_2Cr_2O_7$ on $SrAc_2$** (v. Ba). To (b) add little $K_2Cr_2O_7$ sol.			(c) $SrAc_2$
5. **Ppt. $SrSO_4$ with Am_2SO_4.** To (c) add Am_2SO_4 sol., shake well, let stand, filter, test filtrate.	(2) $SrSO_4$	"	

Supplementary. — (a) Test the Sr flame by dipping a Pt. wire (or a splinter) into a Sr salt, and holding it in the edge of a Bunsen flame. Sep.: (b) Sr and Ba; (c) Group IV and Sr; (d) Group III and Sr; (e) Group II and Sr; (f) Group I and Sr. (g) If HCl were used instead of HAc (in 3), would the subsequent reaction take place just as well? (h) See whether you can ascertain which is more insol., $SrSO_4$ or $BaSO_4$.

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	**NaOH**	$Sr(OH)_2$	white	Acids $60H_2O$	From conc. sol.
2	Am_2CO_3	$SrCO_3$	"	Acids	From neutral or alkaline sol.
3	Na_2CO_3	"	"	"	" " " " " "
4	HNa_2PO_4	$HSrPO_4$	"	M. acids	Partly sol. in AmCl.
5	M_2SO_4	$SrSO_4$	"	$500HCl$, $7000H_2O$	Let it stand some time.
6	Am_2Ox	$SrOx$	"	HCl, HNO_3, AmCl	Slightly sol. in HAc.
7	K_2CrO_4	$SrCrO_4$	yellow	Acids	From conc. sol.
8	Flame test		crimson		Best with $SrCl_2$.

74. CALCIUM, Ca″.

ANALYTICAL REACTIONS.

Directions.	Ppt.	Col.	Sol.
1. Inaction of AmCl and AmOH on Ca salts (v. Mg).			
To sol. add AmCl sol., AmOH.			(a) $CaCl_2$
2. Ppt. $CaCO_3$ (v. Mg).			
To (a) add Am_2CO_3 sol., warm, filter, wash.	(1) $CaCO_3$	white	
3. Dis. $CaCO_3$ in HAc (v. Ba, Sr).			
Pour on (1) a little HAc.			(b) $CaAc_2$
4. Inaction of $K_2Cr_2O_7$ on $CaAc_2$ (v. Ba, Sr).			
To (b) add $K_2Cr_2O_7$ sol.			(c) $CaAc_2$
5. Action of Am_2SO_4 on $CaAc_2$ (v. Sr).			
To (c) add a little Am_2SO_4 sol., very dil.			(d) $CaAc_2$
6. Ppt. Ca with Am_2Ox.			
To (d) add AmOH and Am_2Ox sol.	(2) $CaOx$	"	

Supplementary. — Sep. (a) Ca and Ba; (b) Ca and Sr; (c) Ca and Pb; (d) Ca, Al, and Bi. $CaCO_3$ is the most abundant source of Ca salts. (e) Make several Ca salts from it. (f) Test $CaCl_2$ with a *strong* sol. of Am_2SO_4, and compare with $SrCl_2$. This will show why *very dil.* Am_2SO_4 must be used to sep. Sr and Ca. (g) Test the Ca flame, as in the case of Sr, and compare the two. (h) See whether $CaSO_4$ or $SrSO_4$ is more sol.; (i) $BaSO_4$ or $CaSO_4$.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	NaOH	$Ca(OH)_2$	white	$700H_2O$, AmOH	Solubility decreased by heat. Conc. sol. only.
2	Am_2CO_3	$CaCO_3$	"	Acids	Slightly sol. with AmCl. Neutral or alkaline sol.
3	Na_2CO_3	"	"	"	" " " " "
4	HNa_2PO_4	$HCaPO_4$	"	"	
5	M_2SO_4	$CaSO_4$	"	H_2O (sl.), Acids (sl.), Am_2SO_4	With dil. sol. use C_2H_5OH.
6	Am_2Ox	$CaOx$	"	M. acids	Insol. in HAc, H_2Ox. AmOH aids ppn.

75. MAGNESIUM, Mg''.

ANALYTICAL REACTIONS.

Directions.	Ppt.	Col.	Sol.
1. **Prevent ppn. of $Mg(OH)_2$ and $MgCO_3$** (v. Ba, Sr, Ca). To sol. add AmCl sol., AmOH.			(a) $(AmCl)_2MgCl_2$
2. **Inaction of Am_2CO_3 on $(AmCl)_2MgCl_2$** (v. Ba, Sr, Ca). To (a) add Am_2CO_3 sol.			(b) $(AmCl)_2MgCl_2$
3. **Ppt. $AmMgPO_4$.** To (b) add HNa_2PO_4.	(1) $AmMgPO_4$	white	

Supplementary. — Unless enough AmCl sol. is present, AmOH ppts. half of the Mg as $Mg(OH)_2$. $AmMgPO_4$ will not readily ppt. unless AmOH is present. (a) Verify the above statements. To ppt. $AmMgPO_4$ from very dil. sols., warm gently, stir with a glass rod, and let the sol. stand in a warm place some hours. (b) Verify with dil. sol. (c) Test action of Am_2CO_3 sol. on $MgCl_2$ sol. Does any previous group re-agent ppt. Mg? If so, how may ppn. be prevented? (d) Sep. Mg and Al; (e) Mg and Fe'''; (f) Mg and Ba; (g) Mg and Cu.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	**AmOH**	**$Mg(OH)_2$**	white	**AmCl, 600H_2O**	Only half is ppd., rest is sol. as $Am_2Mg(SO_4)_2$.
2	**NaOH**	"	"	**Acids, AmCl**	
3	**Na_2CO_3**	**$Mg_4(CO_3)_3(OH)_2$**	"	**AmCl**	Var. to $Mg_5(CO_3)_4(OH)_2$. CO_2 liberated.
4	**Am_2CO_3**	**$Am_2Mg(CO_3)_2$**	"	**Dil. HCl**	Variable with strength of sol.
5	**HNa_2PO_4**	**$HMgPO_4$**	"		In conc. sol. only.
6	**HNa_2AsO_4**	"	"		Action is like HNa_2PO_4.
7	**AmCl+AmOH +HNa_2PO_4**	**$AmMgPO_4$**	white cryst.	**Acids, 15,000H_2O**	
8	**B.B.C.C.**		pink		Heat, add drop $Co(NO_3)_2$ sol., heat again; gives pink color, when cool.

76. GROUP V.—Ba, Sr, Ca, Mg.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	SOL.
1. **Prevent ppn. of $Mg(OH)_2$ and $MgCO_3$.** To sol. add AmCl sol., then AmOH.		(a) **$BaCl_2$, $SrCl_2$, $CaCl_2$, $(AmCl)_2MgCl_2$**
2. **Ppt. $BaCO_3$, $SrCO_3$, $CaCO_3$.** To (a) add Am_2CO_3 sol., warm, filter, wash.	(1) **$BaCO_3$, $SrCO_3$, $CaCO_3$**	(b) **$(AmCl)_2MgCl_2$**
3. **Final test for Mg.** To (b) add HNa_2PO_4 sol.	(2) **$AmMgPO_4$**	
4. **Dis. $BaCO_3$, $SrCO_3$, $CaCO_3$ in HAc.** Pour on (1) a little HAc and stir it.		(c) **$BaAc_2$, $SrAc_2$, $CaAc_2$**
5. **Sep. Ba from Sr, Ca.** To (c) add $K_2Cr_2O_7$ sol., filter.	(3) **$BaCrO_4$**	(d) **$SrAc_2$, $CaAc_2$**
6. **Ppt. $SrCO_3$, $CaCO_3$.** Alkalize (d) with AmOH, add Am_2CO_3 sol., boil, filter, wash.	(4) **$SrCO_3$, $CaCO_3$**	
7. **Dis. $SrCO_3$, $CaCO_3$ in HAc.** Dis. (4) in little HAc and dil. it.		(e) **$SrAc_2$, $CaAc_2$**
8. **Sep. Sr and Ca.** To (e) add very dil. Am_2SO_4 sol., shake, let stand 2 hours, filter, test fil.	(5) **$SrSO_4$**	(f) **$CaAc_2$, $CaSO_4$**
9. **Final test for Ca.** Alkalize (f) with AmOH, add Am_2Ox sol.	(6) **CaOx**	

Supplementary.—1. AmOH alone partially ppts. $Mg(OH)_2$. AmCl forms with Mg salts sol. $(AmCl)_2MgCl_2$, on which Am_2CO_3 has no effect. A large excess must be avoided. Mg really forms a group by itself, with a separate re-agent. 4. HAc is used, instead of HCl, as $K_2Cr_2O_7$ sol. ppts. Sr, Ca, as well as Ba from sol. in mineral acids. 5. Only a little of the sol. should first be tested for Ba. If present, the whole must be treated, but if Ba is absent, the rest may at once be tested for Sr (by 8). 5. $BaCrO_4$ must be removed (by repeated filtration if necessary) to prevent interference with Sr test. 6. $SrSO_4$ is slightly sol. in $K_2Cr_2O_7$ sol., hence it is better to re-ppt. Sr and Ca as carbonates, and re-dis. by HAc before adding Am_2SO_4. 8. Very dil. Am_2SO_4 must be used to ppt. Sr, or Ca will be thrown down with it, as $CaSO_4$ is sol. in 400 pts. H_2O. The sol. of Am_2SO_4 should be tested with Ca sol. of a given strength, and if after 2 hours no ppt. forms it is sufficiently dilute. 9. All Sr must be removed before testing for Ca. Make separate sols. of $SrSO_4$, $CaSO_4$, $MgSO_4$. (a) Try $BaCl_2$ sol. with each. (b) Try $SrCl_2$ sol. with each of the last two. (c) Try $CaCl_2$ sol. with the last. Explain the results.

77. SODIUM, Na'.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.
1. **Ppt. Na_2SiF_6** (v. Am, K). To a part of sol. add H_2SiF_6 sol.	(1) **Na_2SiF_6**	white
2. **Inaction of H_2Tr on Na salts** (v. Am, K). To part of original sol. add H_2Tr.	**No ppt.**	
3. **Inaction of H_2PtCl_6 on Na salts** (v. Am, K). To part of original sol. add a few drops of H_2PtCl_6.	"	
4. **Flame test for Na** (v. K). Evap. part of original sol. to dryness, test with Pt wire in outer edge of Bunsen flame. Intercept flame with blue glass (or indigo sol. in t.t.). Hold sol. of $K_2Cr_2O_7$ in t.t. close to Na flame.		deep yellow colorless decolorized
5. **Spectroscopic test for Na.** Test flame as above, using a spectroscope, and compare with a chart (v. Physics).		yellow lines, etc.

Supplementary. — No ppt. is obtained with H_2SiF_6 unless the sol. is strong, hence this is not a very good test. What salts of Na are sol. (v. Table A)? Why should the original sol. be used in each case, *e.g.* in the flame test? Test the action of sol. Na salts on: (a) $AgNO_3$ sol.; (b) $Pb(NO_3)_2$ sol. Since Na salts are sol., explain the results.

78. POTASSIUM, K'.

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.
1. Ppt. K_2PtCl_6 (v. Na). To part of sol. add a few drops of H_2PtCl_6.	(1) K_2PtCl_6	yellow
2. Ppt. HKTr (v. Na). To part of original sol. add H_2Tr sol. and C_2H_5OH.	(2) HKTr	white
3. Ppt. K_2SiF_6 (v. Am, Na). To part of original sol. add H_2SiF_6 sol., warm, let stand.	(3) K_2SiF_6	"
4. Flame test for K (v. Na). Evap. a little of original sol., test with Pt wire in the outer edge of a Bunsen flame. Cover flame with blue glass (or with t.t. of indigo sol.).		red-violet
5. Spectroscopic test for K (v. Na). Examine flame as above, using a spectroscope, and compare with a chart.		blue lines, &c.

Supplementary. — Neither H_2Tr nor H_2SiF_6 will cause a ppt. from dil. sols. What of the common salts of K are insol. (v. Table)? (a) Use several K salts for the above tests, *e.g.* KCl, $KClO_3$, KNO_3, etc. In which is the flame test most distinct? For flame tests use both solids and sols. (b) Dist. by the flame test a mixture of NaCl and KCl. (c) Dist. K and Na by H_2Tr sol.

79. AMMONIUM, NH_4' (or Am).

ANALYTICAL REACTIONS.

DIRECTIONS.	PPT.	COLOR.	SOL.
1. **Ppt. Am_2PtCl_6** (v. Na, K). To a part of the sol. add a few drops of H_2PtCl_6. Evap. and ignite (1).	(1) Am_2PtCl_6 (2) Pt (spongy)	yellow	
2. **Ppt. HAmTr** (v. Na, K). To some original sol. add H_2Tr and C_2H_5OH.	(3) HAmTr	white	
3. **Generate NH_3.** To some of original sol. (or solid) add NaOH sol., heat, and test gas (odor, litmus, HCl).			(a) NH_3 gas
4. **Volatilization.** Heat, and ignite on Pt foil.	No residue		
5. **Nessler's test for NH_3.** To part of original sol. add one or two drops of Nessler's sol.	(4) NHg_2IH_2O	brown	
6. **Inaction of H_2SiF_6 on Am salts** (v. K, Na). To part of original sol. add H_2SiF_6 sol.	No ppt.		

Supplementary. — HAmTr will not ppt. unless a strong sol. is used. Test several compounds of Am by 3 and 4, *e.g.* $AmNO_3$, Am_2Ox, Am_2SO_4, etc.

80. GROUP VI.—Na, K, Am.

Tests.	Results.
1. Test for Na.	
(a) To part of original sol. add H_2SiF_6 sol.	Na_2SiF_6.
(b) Apply flame test with Pt wire.	Yellow flame.
(c) Apply spectroscope with Pt wire.	Yellow lines.
2. Tests for K.	
(a) To part of original sol. add H_2PtCl_6.	K_2PtCl_6.
(b) " " " " " " H_2Tr sol.	$HKTr$.
(c) " " " " " " H_4SiF_6 sol.	K_2SiF_6.
(d) Evap. part of original sol. and try flame test.	Purple flame.
(e) " " " " " " " spectrosc. test.	Blue lines.
3. Tests for Am.	
(a) To part of original sol. add H_2PtCl_6.	Am_2PtCl_6.
(b) " " " " " " H_2Tr and C_2H_5OH.	$HAmTr$.
(c) " " " " " " NaOH sol., warm.	NH_3 gas.
(d) Evap. part of original sol. and ignite.	"
(e) To part of original sol. add a drop of Nessler's sol.	NHg_2IH_2O.

81. BORATES, M_3BO_3, $M_2B_4O_7$.

Tests.	Results.
1. Flame test. Mix a bit of powdered $Na_2B_4O_7$ (or other borate) with a drop or two of H_2SO_4. Dip a Pt wire (or glass rod) in this, and hold it in the edge of a Bunsen flame. The same test may be applied by putting the mixture in an e.d., stirring it with a little C_2H_5OH, and setting the latter on fire; or by mixing a drop or two of glycerin with it and testing with the Pt wire. H_3BO_3 gives a green flame without the use of H_2SO_4.	A green color is imparted to the flame.
2. Turmeric test. Add a *very little* HCl to a sol. of $Na_2B_4O_7$ till it is just acidified, then moisten a piece of turmeric paper with it.	The color of the paper is red-brown. On drying, the color becomes deeper red.

Supplementary. — Phosphates, salts of Cu and of Ba, except $BaSO_4$, also give a green flame, as does C_2H_5Cl (formed by C_2H_5OH and MCl). In testing for B, chlorids should first be removed by $AgNO_3$, and also Cu by H_2S. H_2SO_4 reduces salts of Ba to $BaSO_4$ (which gives no green flame), and the flame of phosphates is a very feeble green. H_3BO_3 in sol. should be evaporated, after first making alkaline to prevent volatilization.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$AgNO_3$	$Ag_3(BO_3)_2$	white		With normal borates. M_3BO_3 also give Ag_2O (brown).
2	$PbAc_2$	$Pb(BO_2)_2$	"		
3	$CaCl_2$	$Ca(BO_2)_2$	"	AmCl, acids	Not ppd. if dil.
4	$BaCl_2$	$Ba(BO_2)_2$	"	" "	" " " "
5	$Al_2(SO_4)_3$	$Al(OH)_3$	"	Acids	
6	$MgCl_2$	$Mg(BO_2)_2$	"	AmCl, acids	

82. NITRATES, MNO_3.

Tests.	Results.
1. Copperas test (v. MNO_3).	
Mix in a t.t. a sol. of MNO_3, or sol. to be tested, with a fresh sol. of $FeSO_4$. *When cold*, introduce into *the bottom* of the tube, with a glass tube used as a pipette, a little H_2SO_4.	$(FeSO_4)_2NO$. Brown ring or halo at junction of liquids.
2. Deflagration test (v. MNO_3, $MClO_3$).	
Heat B.B.C.C. a very little powdered MNO_3; or mix with powdered charcoal, and put into an i.t. enough to cover a penknife point. Heat cautiously.	Vigorous deflagration ensues.
3. Red fumes test (v. MNO_3).	
Put into an i.t. very little MNO_3, heat. Insert a glowing splinter into the tube while heating.	NO_2 and N_2O_3 (red); O (colorless). Splinter burns.
4. Free HNO_3.	
(a) Add a little indigo sol.	Decolorizes, changing to red isatin.
(b) Put a drop on the finger-nail, or dip a feather into it.	Turns animal matter yellow.
(c) Pour a little on Cu and warm it.	Red fumes (NO_2), green liquid, $Cu(NO_3)_2$.

Supplementary. — If a ppt. appears on adding H_2SO_4, let it settle, then decant. H_2SO_4 liberates HNO_3 from nitrates, and $FeSO_4$ reduces HNO_3 *at the surface of contact* to NO, which unites with $FeSO_4$. Let it stand an hour or more if necessary. Heat decomposes this compound $(FeSO_4)_2NO$, expelling NO. The depth of color indicates the strength of nitrate. Heat decomposes all nitrates; from those of the alkali and alkaline earth metals are first formed MNO_2 and O, then M_2O, O, N. Nitrates of Ag and Hg reduce to the metal, others to the oxid only. KNO_3 and $NaNO_3$ show faint fumes, best seen by looking down the tube to a white background. Indigo is also decolorized by Cl, Br, etc. Nitrates of Au and Sb are unknown. All nitrates are soluble except $Bi(NO_3)_3$, which reacts with H_2O and ppts. $Bi(OH)_2NO_3$ (variable), and to a less extent Hg', Hg'', Sn'', Sn^{iv}.

83. NITRITES, MNO_2.

Tests.	Results.
1. Copperas test (v. MNO_3).	
Apply this as in MNO_3.	Same result as in MNO_3, but more readily obtained.
2. Red fumes test (v. MNO_3).	
To a little MNO_2 add a few drops of H_2SO_4.	NO_2 (red fumes) copiously evolved; also HNO_2.
Test the HNO_2 formed with iodo-starch sol.	Starch iodid (blue) formed.
Test it also with $KMnO_4$ sol.	Decolorized.
3. Iodin test.	
Add a few drops of KI sol. to 3 or 4 cc. H_2O, then a few drops of dil. H_2SO_4; finally a little MNO_2 sol. (or substance to be tested).	HI first formed, then H_2O, and I set free by MNO_2 (yellow color).
Test yellow liquid with starch sol.	Iodid of starch (blue).
4. Nitrate test (v. General Reactions).	

Supplementary. — Indigo sol. is decolorized by nitrites, but not by nitrates except by the addition of H_2SO_4, which forms HNO_2. Apply the test. All nitrites are soluble, but $AgNO_2$ and $Pb(NO_2)_2$ only slightly. Hence this test cannot be applied in dil. sols.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$AgNO_2$	$AgNO_2$	white	Acids, H_2O (ex.)	This distinguishes MNO_2 from MNO_3.
2	$Pb(NO_2)_2$	$Pb(NO_2)_2$	"	"	" " " " "

84. CHLORATES, $MClO_3$.

Tests.	Results.
1. **Oxygen test** (v. MNO_2, MNO_3).	
Heat a small quantity of the solid in a t.t.	Melts, O escapes, MCl remains.
Test the O with a glowing splinter.	Re-kindles vigorously.
Dis. the residue when cool, and test for MCl with a drop of $AgNO_3$ sol.	AgCl (white) ppts., sol. in AmOH.
2. **Chlorin test** (v. MNO_2, MNO_3).	
To a *very* little of the powder in a t.t. add with great caution a few drops of H_2SO_4.	ClO_2 (green-yellow, of characteristic odor) liberated with explosive violence.
3. **Deflagration test** (v. MNO_3).	
Powder and heat B.B.C.C.	Deflagrates.
4. **Bleaching test** (v. MNO_2, MNO_3).	
To sol. of indigo add $MClO_3$ sol., and heat.	Turned red (isatin).
Mix sols. of $MClO_3$ and H_2SO_3, and add to indigo sol., without heating.	" " "
5. **Silver nitrate test** (v. MAc, MNO_3).	
To sol. add a few drops of $AgNO_3$ sol.	No effect if the $MClO_3$ is pure.

Supplementary. — All chlorates are soluble. Sb, Sn, and Au have no known chlorates. The deflagration of chlorates is greater than of nitrates. (a) Test this by scratching different kinds of matches. A few drops of HCl added to a few crystals of $MClO_3$, and heated, give ClO_2 and Cl, of characteristic odor. Heat liberates O with violence from chlorates, but the presence of certain oxids (*e.g.* MnO_2) causes it to be liberated more regularly, and at a lower temperature. Dist. (b) $KClO_3$ and KCl; (c) $KClO_3$ and KNO_3; (d) $KClO_3$ and KNO_2.

85. CARBONATES, M_2CO_3.

Tests.	Results.
1. Lime water test.	
To the solid (or sol.) add a little dil. HCl. Test the gas with a drop of $Ca(OH)_2$ sol. on a s.r., or pass it into $Ca(OH)_2$ sol., or shake the lime water with the gas.	CO_2 gas (colorless) is formed, which ppts. white $CaCO_3$ with lime water.
2. Barium hydroxid test.	
Apply this in the same way as the lime water test, using $Ba(OH)_2$ sol. instead of $Ca(OH)_2$.	White $BaCO_3$ falls, ppd. by CO_2 gas, and dissolved with effervescence by HCl.
3. Flame test.	
Put a burning splinter into the gas generated, as above.	The flame is extinguished.

Supplementary. — Any acid (except H_2S, HCy) will act on any carbonate and liberate CO_2, but with varying intensity. H_2CO_3 with a carbonate (*e.g.* Na_2CO_3 or $CaCO_3$) forms the bicarbonate (primary or acid carbonate), as $HNaCO_3$, $H_2Ca(CO_3)_2$. Most carbonates are insol. Heat breaks up many of them, forming the metallic oxid and CO_2. $CaCO_3$ requires a red heat, and the alk. carbonates are not decomposed at the temperature of the Bunsen flame (v. oxalates). Try the action of various acids on carbonates, as: (a) HAc; (b) H_2Ox; (c) H_2Tr. Na, K, Mg, burn in CO_2, liberating C. (d) Pass CO_2 from a gen. into lime water till the ppt. clears, then boil. Dist.: (e) CO_2 and SO_2; (f) CO_2 and H_2S; (g) CO_2 and NO_2; (h) M_2CO_3 and M_2Ox. Some metallic salts with Na_2CO_3 sol. give the primary carbonate, others the secondary, and occasionally a basic salt is formed, as $2PbCO_3.Pb(OH)_2$. Carbonates are white in color.

86. SULFIDS, M_2S.

Tests.	Results.
1. Acid test. To the solid (or sol.) add a little dil. HCl (or H_2SO_4), and warm it. Test the escaping gas (odor, $PbAc_2$ sol. on paper).	H_2S gas formed, with odor of sewer-gas. Black, metallic PbS formed.
2. Coin test. Put a drop of water on a silver coin, and on this a soluble sulfid (if insol., first fuse with Na_2CO_3). (V. M_2SO_3, $M_2S_2O_3$, M_2SO_4.)	Coin is blackened by formation of Ag_2S.
3. Ppn. tests (v. General Reactions).	

Supplementary. — All sulfids, except those of the alkalies and alkaline earths are insol. in H_2O. Most are sol. in dil. acids. A few (FeS_2, HgS, CoS, NiS, etc.) are not acted on by acids, except aqua regia. In such cases H_2S may be made by nascent H, with the addition of a little Zn. No sulfid of Al or Cr is known. Oxidizing agents with sulfids tend to form sulfates, or to liberate S and SO_2. (a) Test several with HNO_3, or aqua regia. Metallic sulfids (including Am_2S) ppt. from sols. metals of the first four groups, except Cr and Al. H_2S ppts. only the first two groups.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Ppt.	Color.	Solvents.	Remarks.
		With H_2S:	With Am_2S:			
1	$Pb(NO_3)_2$	PbS	PbS	black	Hot HNO_3(dil.)	
2	$AgNO_3$	Ag_2S	Ag_2S	"	" " "	
3	$HgNO_3$	Hg_2S	Hg_2S	"	aqua regia	Warming changes to HgS and S.
4	$HgCl_2$	HgS	HgS	"	" "	First white, then yellow, red, black.
5	$BiCl_3$	Bi_2S_3	Bi_2S_3	"	Hot HNO_3(dil.)	
6	$CdCl_2$	CdS	CdS	yellow	" " "	
7	$CuCl_2$	CuS	CuS	brown-black	" " "	
8	$AsCl_3$	As_2S_3	As_2S_3	yellow	Am_2S_2 (ex.), Am_2CO_3	Alkaline sol. not ppd. with H_2S.
9	$SbCl_3$	Sb_2S_3	Sb_2S_3	orange-red	Am_2S_2 (ex.), HCl (hot)	Ppn. with Am_2S is imperfect.
10	$SnCl_2$	SnS	SnS	dark-brown	" "	
11	$FeCl_3$	none	FeS + S	black	Acids	
12	$CoCl_2$	"	CoS	"	aqua regia	AmCl aids ppn.
13	$NiCl_2$	"	NiS	"	" "	" " "
14	$MnCl_2$	"	MnS	pink	Acids	Turns dark-brown. AmCl aids ppn.
15	$ZnCl_2$	"	ZnS	white	"	AmCl aids ppn.

87. SULFITES, M_2SO_3.

Tests.	Results.
1. Acid test. To the solid (or sol.) add a little HCl (or dil. H_2SO_4), and heat it. Test the escaping gas (odor, $K_2Cr_2O_7$ paper).	SO_2 gas is liberated, reducing $K_2Cr_2O_7$ to green $Cr_2(SO_4)_3$.
2. Zinc test. To the original sol. add Zn and dil. H_2SO_4. Test the escaping gas (odor, $PbAc_2$ paper).	H_2S is liberated, forming with $PbAc_2$ black PbS.
3. Coin test (v. M_2S, $M_2S_2O_3$, M_2SO_4). Heat the solid B.B.C.C. *with* Na_2CO_3, also *without*. Put res. on Ag coin on which is a drop of water.	Na_2S is formed, and on the coin black Ag_2S.
4. Oxidation test (v. M_2SO_4). To some of the original sol. add a little euchlorin (or HNO_3), and heat. Test the product with HCl and $BaCl_2$ sol.	M_2SO_3 is oxidized to M_2SO_4, and ppd. with $BaCl_2$ as white $BaSO_4$, insol. in HCl.

Supplementary. — The alkaline sulfites are freely sol. in water. Most others are but slightly sol., or are insol. Sulfites are decomposed by acids (except H_2CO_3, H_3BO_3, H_2S). (a) Ppt. $BaSO_3$ with $BaCl_2$ sol., dis. in HCl, filter if necessary, then oxidize fil. with aqua regia, and try to dis. ppt. in HCl. Heat changes sulfites to M_2O and SO_2; also to M_2SO_4 and M_2S. (b) Make the tests. Sulfites are reducing agents. (c) Test action of SO_2 on $FeCl_3$ sol., then test sol. for Fe″ and Fe‴. (d) Test $HgCl_2$ with SO_2, then test sol. for Hg′ or Hg″. (e) Generate H in a t.t., and test the gas for H_2S with $PbAc_2$ paper, then add a few drops of Na_2SO_3 sol. and test again. (f) Bring together jets of SO_2 and H_2S. (g) Test the bleaching action of SO_2 sol. by putting in it a colored flower, or cambric. (h) Test with litmus sol. (i) Let some SO_2 sol. stand for a few days in an open rec., and apply the $BaCl_2$ and HCl test. (j) Try the action of SO_2 sol. on $CaCO_3$.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$BaCl_2$	$BaSO_3$	white	HCl (dil.)	(V. M_2SO_4.)
2	$AgNO_3$	Ag_2SO_3	"	HNO_3 (dil.)	Boiling ppts. dark-brown Ag.
3	$Pb(NO_3)_2$	$PbSO_3$	"	"	Not blackened on boiling (v. $M_2S_2O_3$).
4	$FeCl_3$	$Fe_2(SO_4)_3$	red		Boiling forms green $FeSO_4$.
5	$HgCl_2$	HgCl	white		Boiling reduces to gray Hg.

88. THIOSULFATES, $M_2S_2O_3$.

Tests.	Results.
1. Bulb-tube test.	
Heat the solid in a tube.	Sublimate of H_2O and S, and liberation of SO_2.
2. Acid test.	
Add to the conc. sol. (or the solid) HCl (or H_2SO_4), warm.	S ppts. (white), SO_2 formed.
Test SO_2 (odor, K_2CrO_4).	
3. Reduction test (v. M_2SO_3).	
(1) To a sol. of starch and KI (made blue by Cl) add a sol. of $M_2S_2O_3$.	Decolorized.
(2) To a sol. of starch and KI (uncolored) add $M_2S_2O_3$ sol. with which a little euchlorin is mixed.	Uncolored, as Cl has oxidized $M_2S_2O_3$ to M_2SO_4 and HCl.
4. Coin test (v. M_2S, M_2SO_3, M_2SO_4).	
Fuse B.B.C.C., put on Ag coin with a drop of H_2O.	Coin is blackened (Ag_2S).
5. Precipitation tests.	
(V. General Reactions.)	

Supplementary. — Thiosulfates, if mixed in a mortar with chlorates, explode violently. They are strong reducing agents. They are mostly sol. in water, except those of Ba, Pb, Ag. Dist.: (a) $M_2S_2O_3$ and M_2S; (b) $M_2S_2O_3$ and M_2SO_3; (c) $M_2S_2O_3$ and M_2SO_4.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$BaCl_2$	BaS_2O_3	white	Dil. acids	Acids ppt. S (white).
2	$AgNO_3$	$Ag_2S_2O_3$	"	$Na_2S_2O_3$ (ex.)	Boiling forms Ag_2S (black). Forms slowly on standing.
3	$PbAc_2$	PbS_2O_3	"	" "	Boiling forms PbS (black). Forms slowly on standing.
4	$HgNO_3$	$Hg_2S_2O_3$	"	" "	Boiling forms Hg_2S (black). Forms slowly on standing.
5	$FeCl_3$	$FeCl_2$			Acts as reducing agent.
6	$CaCl_2$	none			Dist. from M_2SO_3.

89. FLUORIDS, MF.

Tests.	Results.
1. Glass-etching test.	
(1) Put a little powdered CaF_2 into a small lead dish, and mix with enough H_2SO_4 to make a thick paste. Cover the dish with a piece of glass (coated with wax if desired, and a design scratched on it) and let it stand in a warm place for several hours. Remove the wax by melting, and finally with a cloth wet with alcohol, naphtha, or turpentine.	The glass will be etched where exposed to the fumes of HF, SiF_4 being formed.
(2) Mix a little CaF_2 with $HKSO_4$, put into a small t.t., and heat.	The tube will be roughened (or etched) by the HF.
2. Turbidity test.	
Mix a little CaF_2, SiO_2 and H_2SO_4, put into a t.t. and heat it, meantime holding in the tube on a s.r. (or in the loop of a Pt wire) a drop of H_2O.	The water is rendered turbid by the SiF_4 gas, forming H_2SiF_6.
3. Free HF in sol.	
Dip a brush into the liquid in a leaden dish, and mark out a design with it on a glass plate, going over it several times. Avoid getting any HF sol. on the flesh.	The glass will be etched as above.

Supplementary. — Several of the fluorids are soluble. The most important compounds of F are CaF_2 and HF. The latter corrodes glass, porcelain, and all metals except Pb, Pt, and Au. $CaCl_2$ ppts. CaF_2 (transparent, gelatinous). $BaCl_2$ ppts. BaF_2 (white). CaF_2, which occurs in the earth as fluorite or fluorspar and cryolite, Na_3AlF_6, a mineral found in Greenland, are the main sources of F compounds.

90. CHLORIDS, MCl.

Tests.	Results.
1. HCl test.	
Add H_2SO_4 to the solid in t.t., heat. Test gas (odor, color, NH_3).	HCl is liberated.
2. Cl test.	
Mix the solid with MnO_2, add H_2SO_4, heat. Test gas (odor, color, NH_3, indigo paper).	Cl liberated, green-yellow; AmCl with NH_3, indigo paper bleached.
3. Precipitation tests.	
(a) To sol. add $AgNO_3$ sol. Expose part to sunlight, add AmOH to the rest, then HNO_3.	AgCl (white) ppts.; blackens in light; sol. in AmOH; reppd. by HNO_3.
(b) To sol. add $HgNO_3$ sol.; then AmOH.	HgCl (white) ppts.; blackened by AmOH.
(c) To sol. add $Pb(NO_3)_2$ sol. boil with plenty of H_2O.	$PbCl_2$ (white) ppts.; sol. in boiling H_2O.

Supplementary. — Most chlorids are soluble. Except.: Ag, Pb, Hg′, Cu′. $SbCl_3$, $BiCl_3$, $SnCl_2$ decompose water, forming SbOCl, BiOCl, Sn_2OCl_2. H_2SO_4 frees HCl from all chlorids, except HgCl and $HgCl_2$ (and not much from Ag, Pb, Sn chlorids). $HgCl_2$ sol. fails to ppt. $PbCl_2$ from $Pb(NO_3)_2$ sol. (a) Test the last statement. Dist.: (b) MCl and $MClO_3$ (cautiously); (c) MCl and MNO_3. (d) Take 5g $KClO_3$ and divide into 2 parts. Dis. one in H_2O and add $AgNO_3$ sol. Heat the other a few minutes, then dis., and add $AgNO_3$ sol. Compare the results.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	**$AgNO_3$**	**AgCl**	white	**AmOH**, etc.	(v. Exp. 49.)
2	**$Pb(NO_3)_2$**	**$PbCl_2$**	"	**Hot H_2O**	(v. Exp. 48.)
3	**$HgNO_3$**	**HgCl**	"	**HNO_3**	(v. Exp. 50.)

91. BROMIDS, MBr.

Tests.	Results.
1. Bromin test.	
Mix MnO_2 with the solid, add a little H_2SO_4, heat.	Br (red gas) is liberated.
Test the gas by holding in it a paper dipped in starch-paste.	Colors starch yellow to orange.
2. Iodo-starch test.	
Liberate Br as above, and hold in it a paper dipped in a mixture of starch-paste and KI sol.	Br combines with K in KI, liberates I, which forms blue starch-iodid.
3. CS_2 test.	
To the sol. add a drop or two of CS_2 sol., and a drop of euchlorin, and shake it.	Br is liberated and absorbed by CS_2, which is colored yellow.
4. Silver nitrate test.	
To sol. add a drop or two of $AgNO_3$ sol. Try to dis. part of ppt. in HNO_3, another part in AmOH.	AgBr, yellow-white, ppts., insol. in HNO_3, slowly sol. in AmOH.

Supplementary. — Bromids are soluble, except those of Ag, Hg′, Hg″, Cu′, Pb. $BiBr_3$ and $SbBr_3$ need acidulation. Dilute H_2SO_4 acts on bromids to form HBr; strong acid forms Br. (a) To a bromid sol. add euchlorin (very little), and note the color. (b) Dist. MBr and MCl. (c) Pour a little Br vapor into starch-paste, and shake. (d) Mix starch-paste and KBr sol. (very little), and add a drop or two of euchlorin.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Sol.	Remarks.
1	$AgNO_3$	AgBr	pale yellow	AmOH	Only slightly sol. in AmOH.
2	$PbAc_2$	$PbBr_2$	white	Hot H_2O	Less sol. than AgCl in H_2O.
3	$HgNO_3$	HgBr	yellow-white	KBr	

92. IODIDS, MI.

Tests.	Results.
1. Starch test. Mix MnO_2 with the solid, add H_2SO_4, heat, and test with cold starch-paste (v. Exp. 40).	Violet I vapor appears, which colors starch-paste blue.
2. CS_2 test. Add to the sol. a drop of CS_2 [or $(C_2H_5)_2O$], then 2 or 3 drops of euchlorin (avoid excess), and shake it well.	CS_2 dis. the I set free by Cl, and is colored violet at the bottom of the tube. NaOH destroys the color, forming NaI and $NaIO_3$.

Supplementary. — Most iodids are sol., except Pb, Hg′, Hg″, Au. Sb, Bi, Cu, Sn^{iv} iodids need acidulation, as they react with H_2O. The double iodids (KIAgI, etc.) are formed and dis. with excess of KI with salts of Pb, Ag, Hg. I may be liberated from an iodid by Zn and HNO_3, HNO_2 being first formed. Euchlorin or bromin acts the same. Iodids are broken up by oxidizing agents more easily than bromids or chlorids.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$AgNO_3$	AgI	yellow-white	KCy, $Na_2S_2O_3$	Insol. in HNO_3 or AmOH.
2	$Pb(NO_3)_2$	PbI_2	yellow	NaOH (ex.)	Dis. by HNO_3.
3	$HgNO_3$	HgI	green-yellow	$HgNO_3$ (ex.)	" " "
4	$Hg(NO_3)_2$	HgI_2	yellow-red	$Hg(NO_3)_2$ (ex.)	" " "
5	$CuSO_4+H_2SO_3$	CuI	white	H_2O (sl.)	Insufficient reduc. agent ppts. I.

93. ACETATES, $MC_2H_3O_2$ (MAc).

Tests.	Results.
1. Charring test (v. M_2Tr, M_2Ox).	
Heat a very little of the solid in a t.t., take odor.	Melts, expels H_2O, turns black, gives odor of acetone, C_3H_6O (inflammable).
2. Acetic acid test (v. M_2Tr).	
Add to the solid (or sol.) H_2SO_4, heat, take the odor, test with litmus.	HAc is formed, having characteristic odor, and acid reaction.
3. Acetic ether test.	
Add to the sol. a few drops of C_2H_5OH and H_2SO_4, warm, and take odor.	C_2H_5Ac is formed, having characteristic odor.
4. Ferric test.	
To sol. add $FeCl_3$ sol., boil.	$FeAc_3$ (red sol.). Boiling ppts. red-brown basic acetate of Fe‴.
5. Silver nitrate test (v. MNO_3, $MClO_3$).	
To a dil. sol. add $AgNO_3$ sol.	No ppt.
To a strong sol. add $AgNO_3$ sol.	AgAc (white) ppts.
6. Cacodyl test.	
Heat with an alkali and very little As_4O_6; take odor.	Cacodyl oxid $As_2(CH_3)_4O$ evolved (intensely offensive odor).

Supplementary. — All acetates are sol., those of Ag and Hg′ only partially. No acetate of Au is known. Dist.: (a) MAc and M_2Tr; (b) MAc and M_2Ox. (a) Try to make CH_4 in a t.t. by heating dry NaOH and an acetate. (b) Test the combustibility of the gas. (c) Test some vinegar for acetic acid. (d) Make some HgAc; (e) AgAc. (f) See whether HAc will dis. Zn; (g) Fe. (h) If so, test the product for Fe″ and Fe‴.

94. CYANIDS, MCN (MCy).

Tests.	Results.
1. HCy test. Add to sol. a little H_2SO_4 (or HCl). Very cautiously take the odor by fanning the gas to the face with the hand.	HCy is formed (very poisonous); odor of bitter almonds.
2. $Fe(CyS)_3$ test. Put a few drops in an e.d., add equally Am_2S_x, evap. to dryness, dis. in dil. HCl, add $FeCl_3$ sol.	MCyS is formed, sol. in HCl, giving red $Fe(CyS)_3$ sol. with $FeCl_3$.
3. Prussian blue test. To sol. add equally a few drops of NaOH sol. and $FeSO_4$ sol., shake and warm it, add $FeCl_3$. Dis. in dil. HCl (excess).	$FeCy_2$ (sol. in MCy excess), $Fe(OH)_2$, $Fe(OH)_3$ formed, soluble in HCl, but ppg. $Fe_4Fe_3Cy_{18}$, deep blue.
4. $AgNO_3$ test. To sol. add a little $AgNO_3$ sol. Pour off liquid, and boil with HNO_3. If not all dissolves, test liquid with HCl.	AgCy (white) ppts., sol. in hot HNO_3, re-ppd. by HCl.

Supplementary. — Use great care in experimenting with cyanids, as they are very poisonous. Cyanids are insol., except $HgCy_2$ and those of the alkalies and alkaline earths ($BaCy_2$ only slightly). The solutions are alkaline. The ppd. cyanids are often sol. in excess of the cyanids, forming double salts. (a) Make several by ppn., and then dissolve. Dist.: (b) MCy and MAc; (c) MCy and MNO_3. In the Prussian blue test the following equations indicate the order of changes: (1) $2MCy+FeSO_4=FeCy_2+M_2SO_4$; (2) $4MCy+FeCy_2=M_4FeCy_6$; (3) $FeSO_4+2NaOH=Fe(OH)_2+Na_2SO_4$; (4) $2Fe(OH)_2+H_2O+O=2Fe(OH)_3$; (5) $2HCl+Fe(OH)_2=FeCl_2+2H_2O$; (6) $3HCl+Fe(OH)_3=FeCl_3+3H_2O$; (7) $4FeCl_3+3M_4FeCy_6=Fe_4Fe_3Cy_{18}+12MCl$. The vapor of HCy is a deadly poison, NH_3 or Cl inhaled being the antidote. As used in medicine the acid contains only 2% HCy.

95. TARTRATES, $M_2C_4H_4O_6$ (M_2Tr).

Tests.	Results.
1. Ignition test (v. M_2Ox, MAc).	
Heat in i.t. Take odor, note res.	Evolves fumes (white, then brown), CO_2, with odor of burnt sugar, and black residue, M_2CO_3, C.
2. H_2SO_4 test (v. MAc, M_2Ox).	
Heat in i.t. with little H_2SO_4.	CO, CO_2, SO_2, C.
3. Mirror test.	
Add $AgNO_3$ sol. (to a neutral sol.). Almost dis. ppt. with AmOH, then heat gently in t.t.	Ag_2Tr (white) ppts. Mirror of Ag collects on the tube.
4. Acetic acid test (v. MAc, M_2Ox).	
Add to sol. $BaCl_2$ sol. Dis. the ppt. in HAc.	BaOx is sol. in HAc.
Add to sol. $CaCl_2$ sol. Dis. the ppt. in AmCl sol.	CaOx forms, sol. in NaOH, re-ppd. on boiling.
5. Potassium chlorid test.	
Add KCl sol. and C_2H_5OH to the sol., stir with a glass rod.	HKTr (white) ppts. from strong sols. only.

Supplementary. — Only the alkali tartrates are freely soluble, and the bitartrates of the alkali metals are much less so than the normal ones (v. tests for K and Am). Dist.: (a) M_2Tr and M_2Ox; (b) M_2Tr and MNO_2. (c) Make HAmTr. (d) Try the reducing action of H_2Tr on $K_2Cr_2O_7$ sol. (e) See whether CaTr is sol.

96. OXALATES, $M_2C_2O_4$ (M_2Ox).

Tests.	Results.
1. Ignition test (v. M_2Tr, MAc). Heat the solid in an i.t. Test gas with $Ca(OH)_2$ sol., also combustion of CO. H_2Ox decomposes with very slight charring, and without res.	CO_2, CO, M_2O form. On passing the gas through NaOH sol. to remove CO_2, CO burns with blue flame.
2. Acid test (v. MAc, M_2Tr). Heat the solid with H_2SO_4 (which absorbs the H_2O).	CO_2, CO, M_2O form. Dil. HCl, HNO_3 or H_2SO_4 decomposes M_2Ox, forming H_2Ox.
3. Calcium sulfate test. Alkalize the sol. with AmOH, add a little $CaSO_4$ sol. Try to dis. part of the ppt. with HAc, the rest with dil. HCl.	CaOx (white) ppts., sol. in dil. HCl without effervescence, insol. in HAc.

Supplementary. — On igniting oxalates, carbonates and CO first form, then oxids and CO_2. Ag_2Ox and HgOx (which easily decompose with heat) give the metals as a final product. Ag_2O decomposes with explosion. (a) Test several oxalates, first changing them to Ag_2Ox. At moderate temperature H_2Ox sublimes unchanged, except to give off its water of crystallization. Most oxalates are insoluble, except those of the alkali metals and Cr. HCl, HNO_3, or H_2SO_4 forms H_2Ox from its salts. Oxalates are reducing agents, (b) (test with $KMnO_4$), and are almost all white in color. (c) Make several oxalates. (d) See whether H_2Ox will react with metals, *e.g.* Zn and Fe.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.
1	$CaCl_2$	CaOx	white	HCl (dil.)
2	$FeSO_4$	FeOx	yellow-white	
3	$AgNO_3$	Ag_2Ox	white	
4	$Pb(NO_3)_2$	PbOx	"	NaOH, HNO_3

97. SILICATES, M_4SiO_4, M_2SiO_3.

Tests.	Results.
1. Acid test.	
To strong sol. add HCl; then evap. Add HCl, boil, decant; then add to res. NaOH sol., boil.	H_4SiO_4 (gelat., white) formed. Heat reduces to SiO_2, insol. in HCl; partially sol. in NaOH sol.
2. Fusion test (insol. silicates).	
Mix thoroughly 2 pts. dry K_2CO_3, 2 pts. Na_2CO_3, 1 pt. *finely powdered* M_4SiO_4 (*e.g.* slate, etc.), *fuse thoroughly* B.B.C.C. Pulverize, add H_2O, boil, filter. To fil. add HCl, boil, evap. to dryness. Try to dis. in boiling HCl, then in boiling NaOH sol. The SiO_2 may also be tested with CaF_2 and H_2SO_4 in a leaden dish (v. Exp. 28).	K_4SiO_4, Na_4SiO_4, M_2O (M_2CO_3) are formed. First two are sep. by H_2O, and changed by HCl to H_4SiO_4, and by heat to SiO_2 (gritty); insol. in HCl, slightly sol. in NaOH.

Supplementary. — Silicates are insol., except those of the alkalies, which are somewhat soluble. Some are dis. by acids, but many are not. SiO_2 is insol., even in acids (except HF), and is also infusible in the blow-pipe flame, but is fusible in the oxyhydrogen flame. There are two varieties of silica, the amorphous and the crystalline. The former is sol. in NaOH or Na_2CO_3, the latter not. In a bead of Na_2CO_3 at very high temperature, B.B., SiO_2 and all silicates fuse to a clear glass. They will not fuse in a bead of $HNaAmPO_4$. The best fusing mixture contains about half each of Na_2CO_3 and K_2CO_3, the mixture melting at a lower temperature than either alone. Test SiO_2.

98. FERROCYANIDS, M_4FeCy_6.

Tests.	Results.
1. Ferrous test.	
Add to the sol. — acidified with HCl — a few drops of $FeSO_4$ sol.	White $K_2Fe_2Cy_6$ ppts., changing quickly to blue. Darkens by oxidation.
2. Ferric test.	
Add to the sol. $FeCl_3$ sol.	Prussian blue, $Fe_4Fe_8Cy_{18}$, ppts., sol. in H_2Ox (not HCl) to dark-blue liquid. NaOH changes to red-brown $Fe(OH)_3$.
3. Copper test.	
Add to sol. $CuSO_4$ sol.	$Cu_2Fe_2Cy_6$ (chocolate) ppts., insol. in HAc.
4. Silver test.	
Add to sol. $AgNO_3$ sol.	White $Ag_4Fe_2Cy_6$ ppts., insol. in HNO_3 or AmOH. Heated with HNO_3 the ppt. forms $Ag_3Fe_2Cy_6$ (orange-red), sol. in AmOH.

Supplementary. — Ferrocyanids are mostly insol., except those of the alkalies and alkaline earths (Ba slightly). Ferrocyanids represent ferrous compounds, ferricyanids are from ferric salts. The most important of the former is K_4FeCy_6, "yellow prussiate of potash." Make several ferrocyanids by the following table.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$AlCl_3$	$FeCy_2+Al(OH)_3$	white	NaOH	Forms slowly.
2	$SbCl_3$	$Sb_4Fe_3Cy_{18}$	"	"	
3	$BiCl_3$	$Bi_4Fe_3Cy_{18}$	"	"	
4	$CdCl_2$	Cd_2FeCy_6	"	HCl, NaOH	
5	$CaCl_2$	$K_2CaFeCy_6$	"	NaOH	
6	$CoCl_2$	Co_2FeCy_6	green	"	Becomes grad. gray.
7	$CuCl_2$	Cu_2FeCy_6	red-brown	"	
8	$FeSO_4$	$K_2FeFeCy_6$	white-blue	"	Rapidly changes from white to blue.
9	$FeCl_3$	$K_4Fe_3Cy_{18}$	blue	"	
10	$Pb(NO_3)_2$	Pb_2FeCy_6	white	"	
11	$MgCl_2$	$Am_2MgFeCy_6$	"	"	With ammonium salts.
12	$MnCl_2$	Mn_2FeCy_6	"	NaOH, HCl	
13	$HgNO_3$	Hg_4FeCy_6	"	NaOH	
14	$Hg(NO_3)_2$	Hg_2FeCy_6	"	"	Changes to $HgCy_2$ and $Fe_8Fe_2Cy_{12}$ (blue).
15	$NiCl_2$	Ni_2FeCy_6	green-white	"	
16	$AgNO_3$	Ag_4FeCy_6	white	"	Changes to blue.
17	$ZnCl_2$	Zn_2FeCy_6	"	"	

99. FERRICYANIDS, M_3FeCy_6.

Tests.	Results.
1. Ferrous test.	
Add to some of the sol. $FeSO_4$ sol.	Turnbull's blue, $Fe_3Fe_2Cy_{12}$ ppts. insol. in acids. NaOH decolorizes it.
2. Ferric test.	
Add to some of the orig. sol. $FeCl_3$ sol. Dilute if necessary.	No ppt., but dark-green to brown coloration.
3. Copper test.	
Add to some of the orig. sol. $CuSO_4$ sol.	Yellow-green $Cu_3Fe_2Cy_{12}$ ppts., insol. in HCl.
4. Silver test.	
Add to some of the orig. sol. $AgNO_3$ sol.	Orange $Ag_3Fe_2Cy_6$ ppts., sol. in AmOH, not in HNO_3.
5. Zinc test.	
Add to some of the orig. sol. $ZnCl_2$ sol.	Orange $Zn_3Fe_2Cy_{12}$ ppts., sol. in HCl or AmOH.

Supplementary. — Ferricyanids are mostly insol., except those of alkalies and alkaline earths. Alcohol does not ppt. the ferricyanid of Na or K from sol., and this also dist. it from the ferrocyanid. Make the test. K_3FeCy_6 was formerly called red prussiate of potash.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$AgNO_3$	Ag_3FeCy_6	red-brown	AmOH	
2	$BiCl_3$	$BiFeCy_6$	brown		
3	$CdCl_2$	$Cd_3Fe_2Cy_{12}$	yellow	AmOH, HCl	
4	$CoCl_2$	$Co_3Fe_2Cy_{12}$	red-brown	AmOH	Blood-red sol. with AmOH and AmCl.
5	$CuCl_2$	$Cu_3Fe_2Cy_{12}$	yellow-green	"	
6	$FeSO_4$	$Fe_3Fe_2Cy_{12}$	dark-blue	"	
7	$FeCl_3$	none			Green to brown color.
8	$Pb(NO_3)_2$	uncertain	dark-brown	"	In conc. sols. only.
9	$MnCl_2$	$Mn_3Fe_2Cy_{12}$	brown	"	
10	$HgNO_3$	uncertain	red-brown	"	Turns white eventually.
11	$Hg(NO_3)_2$	none			(V. M_4FeCy_6.)
12	$NiCl_2$	$Ni_3Fe_2Cy_{12}$	yellow-green		
13	$ZnCl_2$	$Zn_3Fe_2Cy_{12}$	orange	AmOH, HCl	(V. M_4FeCy_6.)

100. SULFATES, M_2SO_4.

TESTS.	RESULTS.
1. Barium test.	
To a little of the sol. add $BaCl_2$ sol., decant, and to the res. add dil. HCl.	$BaSO_4$ ppts., which is insol. in HCl.
2. Coin test (v. M_2S, M_2SO_3, $M_2S_2O_3$).	
Fuse some of the solid B.B.C.C. (r.f.) with a mixture of Na_2CO_3. Place the product on a silver coin with a drop of water.	Na_2S is formed, which dis. in H_2O and forms black Ag_2S with Ag.
3. Insoluble sulfates.	
Fuse 1 part of the solid with 2 parts each of K_2CO_3 and $NaCO_3$ (in Pt crucible, if no Ag, Pb, or S is present). Dis. what of the product will dis. in H_2O, the rest, separately, in HCl. Test the former for SO_4, the latter for M.	K_2SO_4, Na_2SO_4, M_2CO_3 are formed. The first two are sol. in H_2O, the last in HCl. The former contains the radical, the latter the metal of the original.
4. Free H_2SO_4.	
(1) Put a drop on paper, and evap. it high above a flame, or over a water-bath.	The paper is pulped, rotted, or charred. Comp. with HCl, HNO_3.
(2) Put a few drops on sugar in an e.d. and evap. over a water-bath.	Greenish-black substance is formed.
(3) Evap. some H_2SO_4 nearly to dryness, and add a little C_2H_5OH.	C_2H_4 is liberated, which burns with a luminous flame.

Supplementary. — In the barium test use dil. HCl unless considerable water is present for strong HCl dis. a little $BaSO_4$, and also ppts. $BaCl_2$ from sol. What sulfates are insol.? (a) Prepare some $PbSO_4$, and test it as above in 3. (b) Show that $PbSO_4$ exists in commercial H_2SO_4 by diluting some with its vol. of water. (c) Make $CuSO_4$, and test it. (d) Test Na_2SO_3 for traces of Na_2SO_4. Are most sulfates sol. or insol.?

GENERAL REACTIONS.

	RE-AGT.	PPT.	COLOR.	SOLVENTS.	REMARKS.
1	$BaCl_2$	$BaSO_4$	white	400,000 pts. H_2O	The only salt of Ba (exc. $BaSiF_6$) insol. in dil. HCl.
2	$Pb(NO_3)_2$	$PbSO_4$	"	NaOH, AmAc	C_2H_5OH aids ppn.
3	$SrCl_2$	$SrSO_4$	"	7000 pts. H_2O	Ppts. slowly in dil. sol.
4	$CaCl_2$	$CaSO_4$	"	400 pts. H_2O	No ppt. in weak sol.

101. CHROMATES, M_2CrO_4.

Tests.	Results.
1. Borax bead test. Make a bead of $Na_2B_4O_7$ or $HNaNH_4PO_4$ with Pt wire, dip into sol., fuse in either flame.	Glass, yellow-green when hot, emerald-green when cold.
2. Ppn. tests. Apply the first three tests below (General Reactions).	
3. Hydrogen dioxid test. Acidulate the sol. with H_2SO_4, and add a few drops of dil. H_2O_2 sol. Then add ether, and shake. This will detect 1 part of chromate in 40,000 of water.	A blue compound is formed of uncertain composition, which the ether dis. and carries to the surface as a blue liquid.

Supplementary. — Most chromates are yellow, dichromates are red. Dichromates are confined to the alkali metals. $K_2Cr_2O_7$, etc., ppt. normal chromates, *e.g.* $PbCrO_4$, $BaCrO_4$ from sol. Pb and Ba salts. In both the normal and the dichromates Cr is a hexad, in *ic* salts it is a triad, in *ous* salts a dyad. What other salts are yellow? Acids change chromates to dichromates, alkalies reverse this. Reducing agents (nascent H, SO_2, H_2S) change Cr^{vi} to Cr''', chromate to chromic salts.

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$BaCl_2$	$BaCrO_4$	yellow	Dil. acids	
2	$Pb(NO_3)_2$	$PbCrO_4$	"	HNO_3 (sl.), $NaOH$ (sl.)	Insol. in HAc.
3	$AgNO_3$	Ag_2CrO_4	dark-red	HNO_3 (sl.), $AmOH$	
4	$HgNO_3$	Hg_2CrO_4	"	HNO_3 (sl.)	Ppt. ignited equals Cr_2O_3, Hg, O.
5	$HgCl_2$	$HgCrO_4$	orange	"	
6	$BiCl_3$	$Bi_2(CrO_4)_3$	yellow	"	
7	$CdCl_2$	$CdCrO_4$	"	"	
8	Acids	$M_2Cr_2O_7$	red sol.		Changed back by alkalies to M_2CrO_4 (yellow).
9	H_2S+HCl	$S+CrCl_3$	green sol.		Warming forms SO_2, ppts. $Cr_2O_2CrO_4$ (brown).
10	Am_2S	$Cr(OH)_3$	green	Acids	
11	$C_2H_5OH+HCl$	$CrCl_3$	"		On boiling. Odor of aldehyde.

102. PHOSPHATES, M_3PO_4.

Tests.	Results.
1. **Molybdate test.** To 1 or 2 cc. of sol. add a drop or two of HNO_3, then add 3 or 4 cc. Am_2MoO_4 sol., and let stand.	$Am_3PO_4.12MoO_3$ (var., yel.) ppts., sol. in M_3PO_4 (ex.) or in AmOH.
2. **Magnesium sulfate test** (v. As, Mg). To sol. add AmCl sol., AmOH, $MgSO_4$ sol., gently warm, stir, let stand.	$AmMgPO_4$ (white, crystalline) ppts.
3. **Iron test.** To sol. add a drop of HAc, excess of NaAc sol., and 1 or 2 drops of $FeCl_3$ sol.	$FePO_4$ (yellowish-white, gel.) ppts., sol. in $FeCl_3$ (ex.).
4. **Cobalt test** (v. SiO_2, Al). Heat the solid B.B.C.C., then on white infusible mass put a drop of $CoCl_2$ [or $Co(NO_3)_2$] sol., and heat again.	Residue becomes blue.
5. **Silver nitrate test.** To sol. add $AgNO_3$ sol.	Ag_3PO_4 (yellow) ppts., sol. in HNO_3.

Supplementary. — All phosphates are insoluble except those of the alkali metals; hence sols. of the other metallic salts ppt. phosphates. Most phosphates are somewhat sol. in HCl. If insol. in H_2O, they should be dis. in dil. HCl, AmAc (made from AmOH and HAc) added, then $FeCl_3$ till all the phosphate is ppd. The fil. will now contain a chlorid of the original metal. This test applies specially to phosphates of Ba, Sr, Ca, Mg. (a) Apply the test to each. (b) Test bone-dust in this way. (c) Test samples of fertilizers. Phosphates (and also oxalates) of Ba, Sr, Ca, Mg, if present, may ppt. with Fe, Cr, Al by AmOH. To separate them dis. the ppt. in hot dil. HNO_3, cool and add excess NaOH sol., which ppts. only Fe and the phosphates (or oxalates). Dis. the ppt. in HNO_3, add H_2Tr and AmOH, which in excess ppts. the phosphates and oxalates, dissolving the Fe. (For arseniates and arsenites, v. Exp. 58.)

GENERAL REACTIONS.

	Re-agt.	Ppt.	Color.	Solvents.	Remarks.
1	$Co(NO_3)_2$	$Co_3(PO_4)_2$	red	AmOH, acids	
2	$NiCl_2$	$Ni_3(PO_4)_2$	light-green	" "	
3	$BiCl_3$	$BiPO_4$	white	HCl	Insol. in HNO_3.
4	$Cu(NO_3)_2$	$Cu_3(PO_4)_2$	blue-white	HAc	
5	$HgNO_3$	Hg_3PO_4	white	HNO_3	
6	$Hg(NO_3)_2$	$Hg_3(PO_4)_2$	"	Acids, Am salts	$HgCl_2$ gives no ppt.
7	$BaCl_2$	$Ba_3(PO_4)_2$	"	Dil. HCl	

TABLE A. — Solubilities.

Valence.		Acetate.	Arseniate.	Arsenite.	Borate.	Bromid.	Carbonate.	Chlorate.	Chlorid.	Chromate.	Cyanid.	Ferricyanid.	Ferrocyanid.	Fluorid.	Hydrate.	Iodid.	Nitrate.	Oxalate.	Oxid.	Phosphate.	Silicate.	Sulfate.	Sulfid.	Sulfite.
III	Aluminum	W	A	—	A	W	—	W	W	—	—	—	—	W	A	—	W	A	A	A	A	W	—	A
I	Ammonium	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	—	W	W	W	W	W
III	Antimony (ous)	W	A	A	—	w-A	—	W	w-C	A	—	—	—	W	—	w-A	—	A	A	A	—	w-A	C	A
II	Barium	W	A	w-A	A	W	A	W	W	A	w-A	W	w	a	W	W	W	A	A	A	C	I	W	w-A
III	Bismuth	W	A	—	A	w-A	A	W	w-C	A	w-A	I	c	a	A	w-A	W	A	A	A	—	w-A	c	A
II	Cadmium	W	A	—	A	W	A	W	W	A	A	c	c	w-A	A	W	W	A	A	A	C	W	c	A
II	Calcium	W	w-A	A	A	W	A	W	W	A	W	W	W	a	w	W	W	A	A	A	C	w	W	w-A
III	Chromium (ic)	W	A	—	A	W	—	W	W	A	A	—	—	w-A	A	W	W	W	A	A	C	W	—	A
II	Cobalt (ous)	W	A	A	A	W	A	W	W	A	A	I	I	w-A	A	W	W	A	A	A	C	W	R	A
II	Copper (ic)	W	A	A	A	W	A	W	W	w	A	I	I	w-A	A	w-A	W	A	A	A	C	W	n	A
I	Hydrogen	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W
II	Iron (ous)	W	A	A	A	W	A	W	W	—	A	I	I	w-A	A	W	W	A	A	A	C	W	c	A
III	Iron (ic)	W	A	A	A	W	—	W	W	—	—	W	I	w-A	A	W	W	w-A	A	A	C	W	—	A
II	Lead	W	n	n	n	w-N	A	W	w	n	n	w	N	w-N	n	n	W	n	n	n	C	I	n	n
II	Magnesium	W	A	A	w-A	W	A	W	W	W	W	W	W	a	A	W	W	w-A	A	A	C	W	W	w-A
II	Manganese (ous)	W	A	A	A	W	A	W	W	w	A	I	c	A	A	W	W	A	A	A	C	W	c	A
I	Mercury (ous)	w	n	n	—	N	n	W	I	n	n	n	n	W	—	N	W	n	n	n	—	w	—	n
II	Mercury (ic)	W	A	A	—	w-N	A	W	W	w	A	—	—	W	—	N	W	A	A	A	—	w	R	A
II	Nickel (ous)	W	A	A	A	W	A	W	W	W	A	I	I	w-A	A	W	W	A	A	A	C	W	R	A
I	Potassium	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	—	W	W	W
I	Silver	W	n	n	A	I	n	W	I	N	I	I	W	W	—	I	W	n	n	n	—	w	n	n
I	Sodium	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W
II	Strontium	W	A	w-A	A	W	A	W	W	w	W	W	W	a	A	W	W	A	A	A	w-A	I	W	w-A
II	Tin (ous)	W	A	A	A	W	—	W	w-C	c	—	A	A	W	A	w-A	w-A	A	A	A	—	W	c	—
IV	Tin (ic)	W	A	—	—	W	—	W	W	—	—	—	A	W	A	w-A	w-A	W	A	A	—	W	c	—
II	Zinc	W	A	—	A	W	A	W	W	W	W	c	c	w-A	A	W	W	A	A	A	C	W	c	A

W = soluble in H_2O.
w = sparingly sol. in H_2O.
A = soluble in strong (con.) acids (HCl, HNO_3, $HNO_3 + 3HCl$).
a = soluble in dilute acids (HCl, HNO_3, $HNO_3 + 3HCl$).
w-A = sparingly sol. in H_2O, made sol. by acids.
C = sol. in con. HCl.
c = sol. in dil. HCl.

w-C = sparingly sol. in H_2O. Made sol. by HCl.
N = sol. in con. HNO_3.
n = sol. in dil. HNO_3.
R = sol. in aqua regia ($HNO_3 + 3HCl$).
I = insol. in water or acids.
w-N = sparingly sol. in H_2O. Made sol. by HNO_3.

TABLE B. — Tube Reactions.

Put into a small i.t. (or a bulb tube) a little of the powder, and heat it. Refer the results to this table.

GASES.	SUBLIMATE, ETC.	FUSION.	RESIDUE.	INDICATION.
Red-brown, NO_2				MNO_3, MNO_2
Yellow-gr. (rare) Cl				(MCl)
Red, Br				MBr
Violet, I	Black			MI
Odor, NH_3; HCl tests	White			NH_4
Odor, SO_2; $K_2Cr_2O_7$ test				M_2SO_4, M_2SO_3, $M_2S_2O_3$
Odor, H_2S; $PbAc_2$ test	Yel.-red, brown drops	x		M_2S
Odor, Cy (flame crimson)				(MCy)
CO_2; $Ca(OH)_2$ test				M_2CO_3, M_2Ox
CO (flame blue)				M_2Ox
O (splinter test)				$MClO_3$, MNO_3, MO_2
N_2O " "		x	All finally vaporizes	$AmNO_3$
	Hot: yellow. Cold: white			HgCl
	White	x		$HgCl_2$
White, As_4O_6 (odor)	Cryst. Heat and C = mirror			As_4O_3
	White, needles	x		Sb_2O_3
O (splinter test)	Metallic Hg		Black, changing to red	HgO
	Yel.-red-brown drops	x		S
	Hot: red. Cool: yel.			As_2S_3
	Red when rubbed			HgI_2
	Gray; white if rubbed			Hg
	Black; red if rubbed			HgS
			Black, tarry, burnt sugar odor	M_2Tr
Heavy white	White cryst.			H_2Ox
			Black; odor acetone	MAc
			Hot: red-brown. Cold: light yellow	Bi_2O_3
			Hot: black. Cold: red	Fe_2O_3
			Hot: red-brown. Cold: yellow	PbO
			Hot: yel.-brown. Cold: yellow	SnO_2
			Hot: yel. Cold: white	ZnO
			M_2CO_3, (M_2O), (M)	Org. cpds. of Na, K, Am, Ba, Sr, Ca, Mg
		x	No decomposition	Cpds. of alkalies, + Cl, NO_3 cpds. of alk. earths

TABLE C. — Action with H_2SO_4.

Put a little of the powder into a t.t., and add a *few drops* of H_2SO_4. Unless violent action ensues (as with $KClO_3$) heat it, and observe especially the gas.

Color of Gas.	Odor.	Reference.	Remarks.	Gas.	Indication.
Violet	None	Exp. 92	Dark crystal. sublimate.	I	MI
Yellow	ClO_2	" 84	Substance detonates violently.	ClO_2	$MClO_3$
Red-brown	NO_2	" 82, 83		NO_2, N_2O_3	MNO_3, MNO_2
Red	Br	" 91		Br	MBr
None	HCl	" 90		HCl	MCl
"	Stifling	" 89	Etches glass.	HF	MF
"	H_2S	" 86	In tube open at both ends gives SO_2.	H_2S	M_2S
White	SO_2	" 87	S not liberated.	SO_2	M_2SO_3
"	"	" 88	S is liberated.	"	$M_2S_2O_3$
None	HAc	" 93		HAc	MAc
"	HCy	" 94		HCy	MCy
"	CO	" 96		CO	M_2Ox
"	None	" 10, 101	Kindles splinter.	O	MO_2, M_2CrO_4
"	"	" 85	White ppt. with lime water.	CO_2	M_2CO_3

TABLE D. — Heat on Charcoal (B.B.C.C.).

With the round end of the forceps make a slight depression in a piece of charcoal, and put in a little of the powdered substance. With a mouth blowpipe blow the flame against it, cautiously at first, afterwards strongly.

Results.	Indications.
1. **Substance melts and runs into the coal.**	Salts of the alkalies, and some alkaline earths
2. **Leaves white, infusible residue.**	
Add to this a drop of $CoCl_2$ sol., and heat again. The color may be:	
Blue,	**Al, SiO_2, M_3PO_4**
Green,	**Zn**
Pink.	**Mg**
3. **Coats charcoal:**	
White, volatile, garlic odor, no globule.	**As**
" " hard, brittle globule.	**Sb**
" when cool, yellow when hot, no globule.	**Zn**
" " " " " " globule malleable, but difficultly formed.	**Sn**
Yellow, globule easily formed, fusible, soft, malleable.	**Pb**
Yellow (orange when hot), globule hard, brittle.	**Bi**
Brown or red, no globule.	**Cd**
4. **Deflagrates.**	**$MClO_3$, MNO_3**
5. **Volatilizes.**	**Am**
6. **Gives odor.**	
Choking (SO_2).	**S or M_2S**
Garlic.	**As**

TABLE E.

Heat on Charcoal with Na_2CO_3 (B.B.C.C., Na_2CO_3).

Blow the inner flame (r.f.) for some time against a *mixture* of the powdered substance, and dry, fine Na_2CO_3, using charcoal, as in Table D.

Metallic Globule.	No Globule.	Infusible Powder.	Colored Mass.	Solid Residue.	Incrustation.	Indication.
x				White, brilliant, malleable, non-oxidizable.	In strong o.f. red-brown.	Ag
x				Hard, brilliant, oxidizable, reddish-white.	B.B. volatile. Hot, orange; cold, lemon-yellow.	Bi
x				Red, malleable; oxidizes to black; green flame.		Cu
x				Easily formed, fusible, malleable, oxidizes to yel.	Hot, lemon-yellow; cold, sulfur-yellow; vol. B.B.; O flame blue.	Pb
x				White, malleable, difficultly formed, easily oxidized.	Hot, yellow; cold, white near assay; non-vol.	Sn
	x				White, volatile; light-blue flame; garlic odor.	As
	x				Red-brown (like peacock feather); vol. B.B.	Cd
x	(x)			White, brittle, oxidizable.	Hot, yel.; cold, blue-white; vol. B.B. pale green.	Sb
	x				Hot, yellow; cold, white. With $Co(NO_3)_2$ green.	Zn
		x		Infusible magnetic powder, white, gray, black.		Fe
		x		" " " " "		Co
		x		" " " " "		Ni
			x	Yellow mass.		Cr
			x	Green "		Mn

TABLE F. — Flame Reactions.

Fuse one end of a Pt wire into a short glass tube. Hold the wire in the outer edge of a Bunsen flame till the latter loses the yellow color at first imparted. Moisten the wire with H_2O or HCl, and dip it into a little of the salt, then hold it again in the flame, noting the color. Clean the wire with HCl and the flame.

Flame Color.	Remarks.	Indication.
1. Violet	Seen through blue glass looks red-violet; otherwise Na obscures it.	**K**
2. Bluish		**As**
3. Blue		**Pb**
4. Blue-green	Moistened with H_2SO_4.	**M_3PO_4**
5. "	First blue, then green.	**Cu, espec. $CuCl_2$**
6. Yellow-green	Moistened with H_2SO_4.	**M_3BO_3**
7. "	" " HCl.	**Ba**
8. Greenish	Very slight color.	**Sb**
9. Bronze-green	" evanescent.	**MNO_3**
10. Orange-yellow	Blue glass obscures the color.	**Na**
11. Orange-red	Obscured by Ba.	**Ca**
12. Crimson	" " "	**Sr**

TABLE G. — Bead Reactions.

Make a loop in the end of a Pt wire (Fig. 38, Exp. 45), moisten and dip it into some powdered borax, then hold it in the flame; repeat till you have a glass bead filling the loop. Test a weak sol. (or the *smallest bit* of powder) by bringing the bead in contact with it, then blowing the flame against it with a blowpipe.

Oxidizing Flame (o.f., outer).		Reducing Flame (r.f., inner).		Indication.
Hot.	*Cold.*	*Hot.*	*Cold.*	
Blue	Blue	Blue	Blue	**Co**
Yellow-red	Green	Green	Green	**Cr**
Green	Blue-green	Colorless	Red-brown	**Cu**
Yellow-red	Yellow	Bottle-green	Bottle-green	**Fe**
Violet	Violet	Colorless	Colorless	**Mn**
"	Red-brown	Gray, cloudy	Gray, cloudy	**Ni**

TABLE H. — Acid Radicals — Résumé.

SALTS.	$BaCl_2$.	$BaCl_2$+HCl.	$AgNO_3$.	$AgNO_3$+HNO_3.	OTHER RE-AGENTS AND RESULTS. For further explanation, v. Exps. 81-102. — Abbreviations: b. black, bl. blue, br. brown, ch. chocolate, d. dark, g. green, l. light, o. orange, r. red, v. violet, w. white, y. yellow.
M_2SO_4	w.	w.	(w.)		$Pb(NO_3)_2$: w. $SrCl_2$: w. B.B.C.C. with Na_2CO_3 on Ag: b.
M_2SO_4	w.		w.		$BaCl_2$ + HCl + Cl: $BaSO_4$. HCl: SO_2 [odor, $K_2Cr_2O_7$: g.].
$M_2S_2O_3$	w.		w.		$BaCl_2$+HCl: S. HCl: S+SO_2 [odor, $K_2Cr_2O_7$: g.].
M_2CO_3	w.		w.		HCl: CO_2 [$Ca(OH)_2$: w.]. Heat: (M_2O, CO_2).
M_2CrO_4	l.-y.		d.-r.		$Pb(NO_3)_2$: y. $HgNO_3$: d.-r. $HgCl_2$: o. $BiCl_3$: y. $Cd(NO_3)_2$: y. H_2S: y. Acids: r, which y. by alk.
M_3PO_4	w.		l.-y.		AmCl+AmOH+$MgSO_4$: w. HNO_3+Am_2MoO_4: y. B.B.C.C.+$Co(NO_3)_2$: bl.
M_3AsO_4	w.		ch.-br.		H_2S, warm: y. AmCl + AmOH + $MgSO_4$: w. B.B.C.C.: As [garlic odor, white fumes].
M_3AsO_3	w.		y.-w.		H_2S+HCl: y. $CuSO_4$ (neutral): g. K_2CrO_4+heat: g. $KMnO_4$: decol. C.C.+heat, t.t.: mirror.
M_3BO_3	w.		w.		H_2SO_4, Pt wire: green flame. Turmeric paper + H_3BO_3: br. H_2SO_4+C_2H_5OH: green flame.
M_2Ox	w.		w.		$SrCl_2$: w. $CaCl_2$: w. [sol. in HCl, insol. in HAc]. Heat: CO, CO_2, M_2CO_3, M_2O or M, no charring. H_2SO_4: effervesc., CO_2.
M_2Tr	w.		w.		$CaCl_2$: w. [sol. in NaOH]. KCl: w. Heat: chars [odor]. $AgNO_3$+AmOH: Ag mirror.
M_4SiO_4	w.		l.-y.		HCl+heat: SiO_2. Na_2CO_3 bead: effervesc., forms glass. $HNaAmPO_4$ bead: insol. B.B.C.C.+HCl, evap.: SiO_2, sol. in NaOH.
MNO_2	w.		w.		H_2SO_4: NO_2. HAc+$KMnO_4$: decol. HAc+$AgNO_3$: w. HAc+KI+starch: bl.
MF	w.				H_2SO_4+heat: etches glass, makes H_2O turbid. $HKSO_4$+heat: green flame.
MCl			w.	w.	H_2SO_4: HCl. H_2SO_4+MnO_2: Cl. $AgNO_3$: w. [sol. in AmOH, action of light]. $Pb(NO_3)_2$: w.
MBr			y.-w.	y.-w.	$PbAc_2$: w. CS_2 + Cl: y. H_2SO_4 + MnO_2: Br [starch, KI+starch].
MI			y.-w.	y.-w.	$Pb(NO_3)_2$: y. $HgNO_3$: y.-g. $HgCl_2$: y.-r. CS_2+Cl: v. H_2SO_4+MnO_2: I [starch test].
MCy			y.-w.	y.-w.	$CuSO_4$: y.-g. $Co(NO_3)_2$: w.-br. $NiCl_2$: y.-g. H_2SO_4: HCy [odor cautiously]. KOH+$FeSO_4$ +HCl: bl.
M_2S			b.	b.	HCl: H_2S [odor, $PbAc_2$]. B.B.C.C. on Ag: b.
M_4FeCy_6			w.	g-br	$FeCl_3$: bl. $FeSO_4$: bl.-w. $CuSO_4$: br.-r.
M_3FeCy_6			o.	o.	$FeCl_3$: r.-br. $CuSO_4$: g.-y.
MNO_3					Heat, i.t.: O, NO_2. B.B.C.C.: deflagrates. H_2SO_4: NO_2. $FeSO_4$ + H_2SO_4: r.-br. H_2SO_4 + indigo sol.: decol.
$MClO_3$					HCl+heat: g.-y. Heat, i.t.: O. H_2SO_4 (very little): Cl_2O_4 [y. gas].
MAc					Heat: blackens [odor]. H_2SO_4: HAc [odor]. H_2SO_4 + C_2H_5OH: C_2H_5Ac [odor]. $FeCl_3$, boil: r.

TABLE I. — Acid Radicals.

Arranged according to convenient method of detection.

1. **Flame** { H_3BO_3 ($M_3BO_3 + H_2SO_4$).
2. **Gas** { (with heat) { MNO_3.
   (with acid) { MNO_2, $MClO_3$, M_2CO_3, M_2S, M_2SO_3, $M_2S_2O_3$.
   (with heat and acid) { MF, MCl, MBr, MI, MAc, MCy.
3. **Residue** { (with heat) { M_2Tr, M_2Ox.
   (with heat and acid) { M_4SiO_4 (M_2SiO_3).
4. **Precipitate** { (iron-cyanids) { M_4FeCy_6, M_3FeCy_6.
   (insoluble) { M_2SO_4.
   (unclassed) { M_2CrO_4, M_3PO_4, M_3AsO_4, M_3AsO_3.

COMPLETE ANALYSIS.

In making a *complete analysis*, *i.e.* analysis for the metal and acid radical, the following order should be observed, and the pupil should begin with only a single substance, such as $Pb(NO_3)_2$, $FeCl_3$, etc., and should make a careful record under each head.

1. **External properties**: Color, luster, form (crystalline or amorphous), taste, odor, deliquescent, efflorescent, etc.
2. **Heat in glass tube** (v. Table B).
3. **Heat with H_2SO_4** (v. Table C).
4. **Heat B.B.C.C.** (v. Table D).
5. **Heat B.B.C.C. Na_2CO_3** (r.f.) (v. Table E).
6. **Apply flame test** (v. Table F).
7. **Apply Borax bead test** (v. Table G).
8. **Test solubility** (v. Table A).
9. **Test for Metals** (aqueous analysis) (v. Exps. 48–80).
10. **Test for Acid radicals** (v. Tables H, I).
11. **State conclusion as to composition.**

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